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Editor

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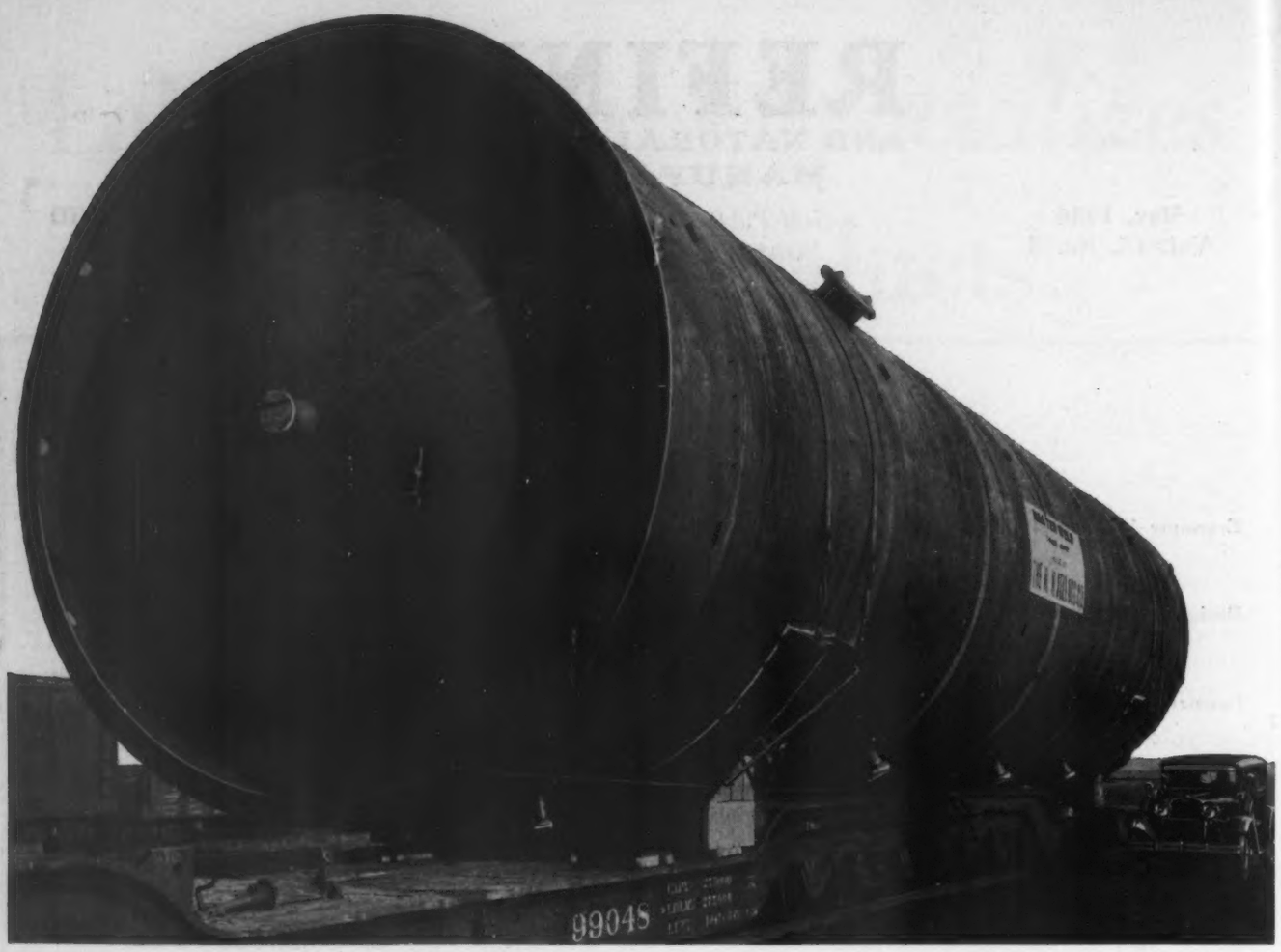

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VOLUME 15, No. 5 MAY, 1936

REFINER
AND NATURAL GASOLINE MANUFACTURER

A GULF PUBLISHING COMPANY PUBLICATION

The Look Box

THE greatest display of new and improved equipment for refining and natural gasoline manufacturing operations in the history of the International Petroleum Exposition is assembled at this year's show at Tulsa.

International Petroleum Exposition

The opening day, Saturday, May 16, is Refiners' day, with W. G. Skelly, president of the exposition, as host, while Sunday, May 17, is Natural Gasoline Manufacturers' day, with W. K. Warren as official host.

The Tulsa Junior Chamber of Commerce is in charge of the plant visit for refiners and natural gasoline manufacturers, and this group will inspect the large, modern refinery of the Mid-Continent Petroleum Corporation at West Tulsa. The annual safety meet, always an interesting feature of the exposition, includes first-aid teams from refineries throughout the Mid-Continent.

Probably the outstanding feature of the hundreds of exhibits and demonstrations in the Scientific and Technical Building is the complete road oil and asphalt testing laboratory. Prominent asphalt and road oil technicians are in charge and give frequent demonstrations of the methods used in the laboratory. The equipment used includes the latest viscosity and ductility machines, a float test, an emulsifying machine, and other laboratory apparatus.

Other scientific apparatus exhibited includes a super-sensitive photoelectric ray, a synchronizing remote-control unit, a remote-control level indicator and recorder, a colorimeter which analyzes color from clearest Saybolt to total black; an electronic temperature control said to be capable of controlling to an accuracy of .001 degree, a cathode ray oscilloscope for analyzing frequency characteristics and watching and isolating vibration, as well as apparatus for obtaining viscosity, adhesion, cohesion, oiliness, surface tension, etc.

In addition to the Scientific and Technical Building, 180,000 square feet of exhibit space is devoted to displays by manufacturers of equipment. Due to exhibits of heavy machinery used in drilling operations, including three drilling rigs actually "making hole," the production department leads all other branches of the industry in the amount of space occupied, but the refining and natural gasoline branch run it a close second, with marketing third.

In order that those not able to attend the International Petroleum Exposition this year may secure a fair picture of the "quality and quantity" of equipment and process displayed. THE REFINER is devoting a large special section elsewhere in this issue to the presentation of descriptive matter, photographs and drawings of

scores of items of equipment found at the exposition.

While many exhibitors are withholding details of new and improved equipment which is shown this year, a large number have provided advance information, and among the hundreds of new and improved equipment items, processes and methods pertaining to the manufacturing branch of the industry are the following: Scores of new pipe cutting and threading tools and machines of all types and sizes; thermometers with mercury column showing red, although the mercury itself is not colored; a new boiler water-level control device; explosion-proof unilets for wiring in hazardous locations; pressure filters for Diesel engine lubricating oil; a new burner for utilization of fixed gases and heavy refinery residual oils as fuel; a new type distillation unit for natural gasoline plants; a balloon for prevention of evaporation on groups of storage tanks, and new styles and sizes of completely enclosed and felt-sealed and shielded thrust bearings.

Still other exhibitors are showing such items as a new light-weight cellular insulating refractory concrete; steam traps of greater capacities; a wide variety of new safety devices and equipment, including a head band for safety goggles that stretches but contains no rubber; a new check valve design combining union and valve; new meters and controllers designed to meet "sour" gas and high pressure conditions; new chemical measuring pumps for injecting chemicals into oil lines, and similar services; a combustible gas indicator for testing explosive gas hazards; improved methods of machine-applied pipe coatings; improved tube cleaners, making for less "time-out" in cleaning stills, and return bend fittings for heating coils for use in polymerization and hydrogenation, improved in construction and design.

Further exhibits having to do with the manufacturing branch of the industry include a refractory coating material which is sprayed on burned-out fire brick; tellurium chemical sheet lead which toughens under vibration; a potentiometer stabilog which is equipped with a chart and pen to record control deviations; a new remote valve control which permits accurate manual adjustments on distant air-operated valves from a central panel board; improved designs in gas analyzers and meters for the scientific burning of fuel; a new super-fractionator which makes possible for the first time high fractionating efficiency and great capacity in an apparatus of centrifugal pump dimensions; pyrometers for measuring Diesel engine exhaust temperatures; new air-operated equipment for full automatic control of temperature, pressure, flow and liquid level; condenser and exchanger tubes made from new alloys of greater strength; a number of new fire-fighting devices using carbon dioxide gas; new steam-cleaning methods for large equipment, and the latest equipment for paint-stripping

(Continued on page 71a)

Exposition grounds at Tulsa

Economy in Natural Gasoline Plant Operation

W. L. NELSON, *Consultant*
Professor of Petroleum Refining
University of Tulsa

IN the operation of most natural gasoline plants, the practice is to absorb as large a percentage of the pentane and heavier hydrocarbons as possible. Operation is assumed to be most profitable when the residue gas contains no pentane-plus hydrocarbons. Under certain economic conditions such as a very high gasoline price or a very low operating cost, the absorption of 99 percent of the pentane-plus hydrocarbons is advantageous, but usually these conditions are such that the absorption of 90 to 98 percent of the available pentane-plus hydrocarbons is a more profitable operation. In some instances even a lower percentage absorption is advisable.

The major reason for stating that all of the heavy hydrocarbons should not be absorbed, is the tremendously larger amount of oil that must be recirculated if 99 percent of the pentanes are to be recovered. As an example, the amount of oil that must be recirculated for different recoveries of pentane are about as follows:

Recovery of Pentane-Plus, Percent of that in Gas	Gallons of Absorption Oil, Based on 5 Gallons for Recovering 60 Percent
60	5
70	6
80	7.25
90	9
95	11
99	16
99.5	18

Thus to increase the recovery from 95 to 99 percent increases the oil recirculation by 50 percent, or to increase the recovery from 60 to 99.5 percent requires that the oil recirculation be increased by about 360 percent.

EQUATION OF PROFIT

The source of all profit for the plant is the gasoline that is produced—and in order to handle this factor mathematically it is necessary to derive an equation of yield of gasoline as a function of the pentane-plus in the gas and if possible as a function of some property such as the vapor-pressure or grade of gasoline. Such an equation was derived from data on Mid-Continent gasoline which were published by Alden in the Oil & Gas Journal, May 21, page 74, 1931.

$$Y = 0.0037BR\ (1.097 - 0.025V + 0.00153V^2) = 0.0037BRy \quad (1)$$

in which

Y = yield of gasoline as a function of the volume percentage of pentane-plus recovered from the gas, gal. per 1000 ft³ of gas.

B = pentane-plus in gas, gas volume percent.

R = recovery of pentane-plus, percent.

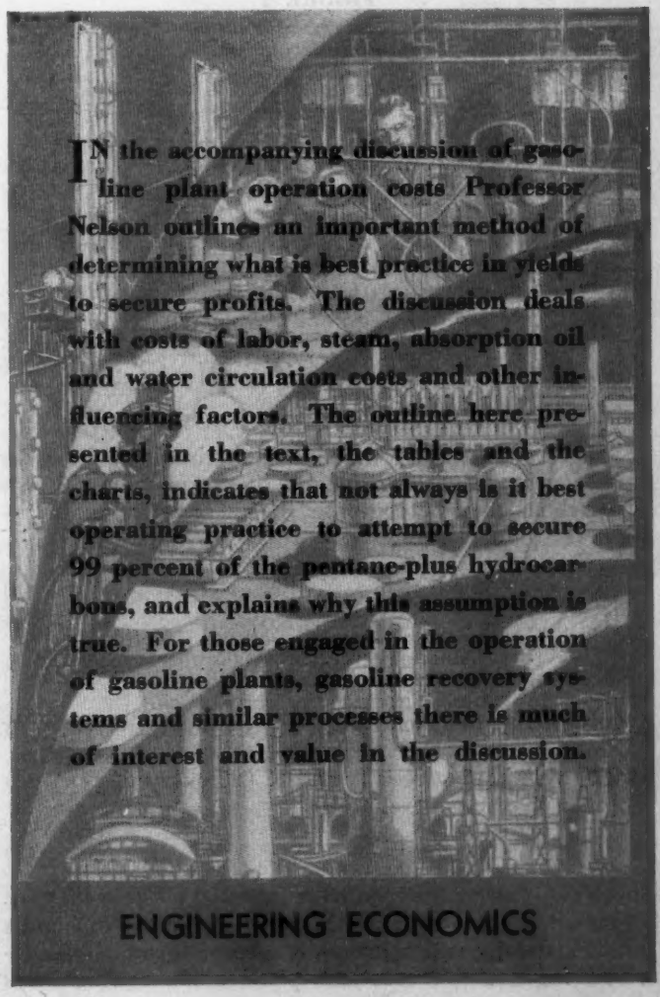
V = vapor-pressure of finished gasoline.

y = gallons of finished gasoline per gallon of pentane-plus.

The data from which Equation (1) was derived is given below. The percentages of pentane-plus and butane were based on a pentane ratio of 0.47.

GRADE	Butane, Percent	Pentane+, Percent	Finished Gasoline per Gallon of Pentane+
10–40	0	100	1
14–55	6.5	93.5	1.069
18–55	14	86	1.163
22–70	22.5	77.5	1.29
26–70	31	69	1.45
30–70	42	58	1.725

The value of the gasoline that is produced can be obtained by multiplying Equation (1) by C_g which is the current market value of gasoline. Although C_g is a function of the grade of gasoline, the range of price is not great, and to use a constant value of C_g does not


IN the accompanying discussion of gasoline plant operation costs Professor Nelson outlines an important method of determining what is best practice in yields to secure profits. The discussion deals with costs of labor, steam, absorption oil and water circulation costs and other influencing factors. The outline here presented in the text, the tables and the charts, indicates that not always is it best operating practice to attempt to secure 99 percent of the pentane-plus hydrocarbons, and explains why this assumption is true. For those engaged in the operation of gasoline plants, gasoline recovery systems and similar processes there is much of interest and value in the discussion.
ENGINEERING ECONOMICS

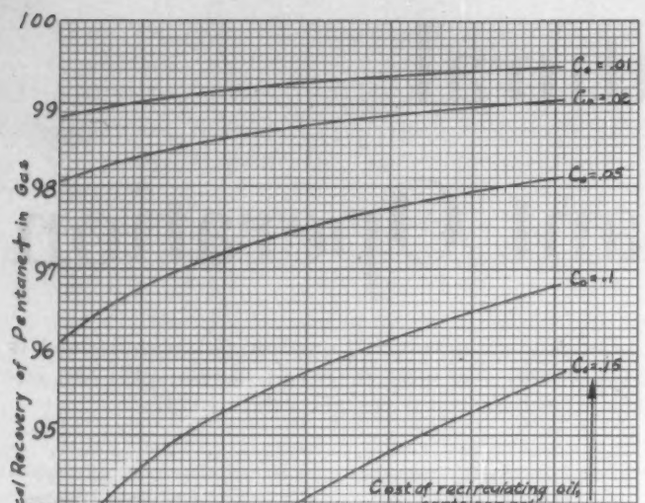


FIGURE 1

Most economical recovery of Pentane, versus cost of oil recirculation.

introduce an error unless the economic recovery is compared for several grades of gasoline.

The profit from the plant is equal to the value of the gasoline less the costs of operation, thus:

Profit = Value of gasoline minus
- (1) Cost to recirculate oil through absorber and stripper.
- (2) Cost of stabilizing the gasoline.
- (3) Costs that are substantially independent of the amount of pentane recovered, such as depreciation, labor, supervision, repairs, laboratory, etc.

AMOUNT OF OIL RECIRCULATION

The amount of oil that must be circulated in order to recover a particular amount of the pentane-plus hydrocarbons may be computed by means of the absorption factor of Kremser or Brown:

$$G = \left(\frac{KM}{3.156d}\right) A \qquad (2)$$

in which, G = gallons of absorption oil per 1000 ft.³
K = equilibrium constant at absorber temperature.
M = mol weight of absorption oil.
d = specific gravity of absorption oil.
A = absorption factor — a function of the recovery of pentane-plus.

The relation between A and R (recovery of pentane) is given by the general form —

$$R = \frac{A^{n+1} - A}{A^{n+1} - 1} \qquad (3)$$

in which, n = number of theoretically perfect plates in absorber.

However, this equation is too complicated to handle mathematically and hence an empirical formula was derived for a six theoretical plate column which was thought to be a reasonable number of plates for current practice.

$$R = \frac{100\,A^{3} - 13.5}{A^{3}} \qquad (4)$$

or——

$$A = \left(\frac{13.5}{100\text{-}R}\right)^{0.33} \qquad (5)$$

This equation fits the data reasonably well through the range of 75 to 99 percent recovery. Equation (2) then becomes:

$$G = \frac{(13.5)^{0.33} KM}{3.156d(100\text{-}R)^{0.33}} \qquad (6)$$

Thus, the cost of oil recirculation may be introduced by means of Equation (6).

The second operation-cost item, the cost of stabilization, is a function of the amount of gasoline produced, or of Equation (1).

The profit equation then assumes the following mathematical form

$$P = 0.0037BR\ (1.097 - 0.025V + 0.00153V^2)C_g - \left[\frac{(13.5)^{0.33} KMC_o}{3.156d}\right] \frac{1}{(100\text{-}R)^{0.33}} - 0.0037BR\ (1.097 - 0.025V + 0.00153V^2)C_s - C_d \qquad (7)$$

in which, P = profit, cents per 1000 ft.³
C_g = value of gasoline, cents per gallon.
C_o = cost of oil circulation, cents per gallon.
C_s = cost of stabilization, cents per gallon of gasoline.
C_d = fixed operating costs.
y = $(1.097 - 0.025V + 0.00153V^2)$.

or——

$$P = \left[0.0037B(1.097 - 0.025V + 0.00153V^2)(C_g - C_s)\right] R - \left[\frac{2.38\,KMC_o}{3.156d}\right] \frac{1}{(100\text{-}R)^{0.33}} - C_d \qquad (8)$$

By letting m and n represent the two large constants

$$P = mR - \frac{n}{(100 - R)^{0.33}} - C_d \qquad (9)$$

The value of R that will produce a maximum profit can be obtained by differentiating Equation (9) and setting the differential form equal to zero, or

$$\frac{dP}{dR} = O = m - \frac{0.33n}{(100 - R)^{1.33}}$$

$$O = m\,(100 - R)^{1.33} - 0.33n$$

and——

$$R_g = 100 - \left(0.33\frac{n}{m}\right)^{0.75} = 100 - 0.472\left(\frac{n}{m}\right)^{0.75} \qquad (10)$$

The constants are thus grouped into the term n/m, which may be simplified to some extent by inserting some of the constants

$$\frac{n}{m} = \frac{(13.5)^{0.33} MK\,C_o}{3.156 \times 0.0037Bd(C_g - C_s)(1.097 - 0.025V + 0.00153V^2)}$$

$$= \frac{2.38 \times 190}{3.156 \times 0.0037 \times 0.84} \times \frac{KC_o}{By(C_g - C_s)}$$

in which, M = 190
d = .84
y = the yield of finished gasoline per gallon of pentane-plus.

$$R_g = 100 - 0.472\left(\frac{2.38 \times 190}{3.156 \times 0.0037 \times 0.84}\right)^{.75}\left(\frac{KC_o}{By(C_g - C_s)}\right)^{.75}$$

$$R_g = 100 - 1{,}500\left(\frac{KC_o}{By(C_g - C_s)}\right)^{.75} \qquad (11)$$

This is a general form that is applicable to all six-

plate absorbers. The constants in the three-fourths power expression can all be evaluated for a particular plant and thus permit the calculation of the most economical pentane-plus recovery.

For a low-pressure absorber (50 pounds absolute) the general equation is.

$$R_n = 100 - 531\left(\frac{C_o}{B_y\,(C_g - C_s)}\right)^{.75} \qquad (12)$$

and for a high-pressure absorber (400 pounds absolute)

$$R_n = 100 - 224\left(\frac{C_o}{B_y\,(C_g - C_s)}\right)^{.75} \qquad (13)$$

VALUE OF THE COST CONSTANTS

The values to use for C_o, C_g, and C_s (the cost of recirculating oil, the value of finished gasoline, and the cost of stabilizing the gasoline respectively) can be obtained most easily by a study of the cost data of the particular plant that is being considered. However, in order to make an analysis of the effect of the several variables, on the most economic pentane recovery, an attempt has been made to evaluate reasonable values for the cost factors.

The cost of recirculating absorption oil is not as great as it might seem at first glance. The cost of generating steam for pumping and handling the oil and for stripping the oil in the still is often very low because the fuel may be worth almost nothing. Furthermore, the operating labor is almost independent of the amount of oil recirculation because the plant and the boilers must be

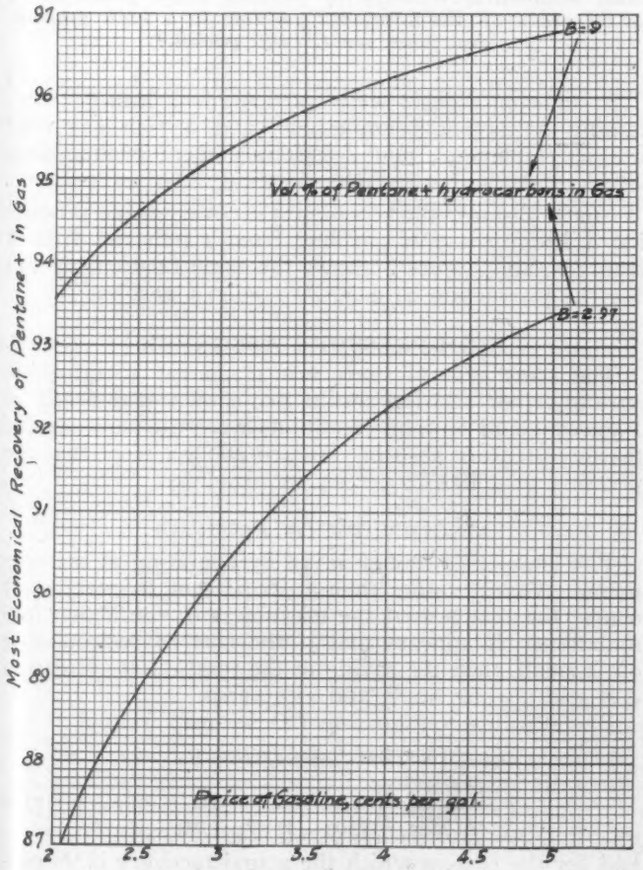


FIGURE 2

Most economical recovery of pentane when producing a 14-55 grade of Mid-Continent Gasoline in a low pressure absorption plant. (50 pound.)

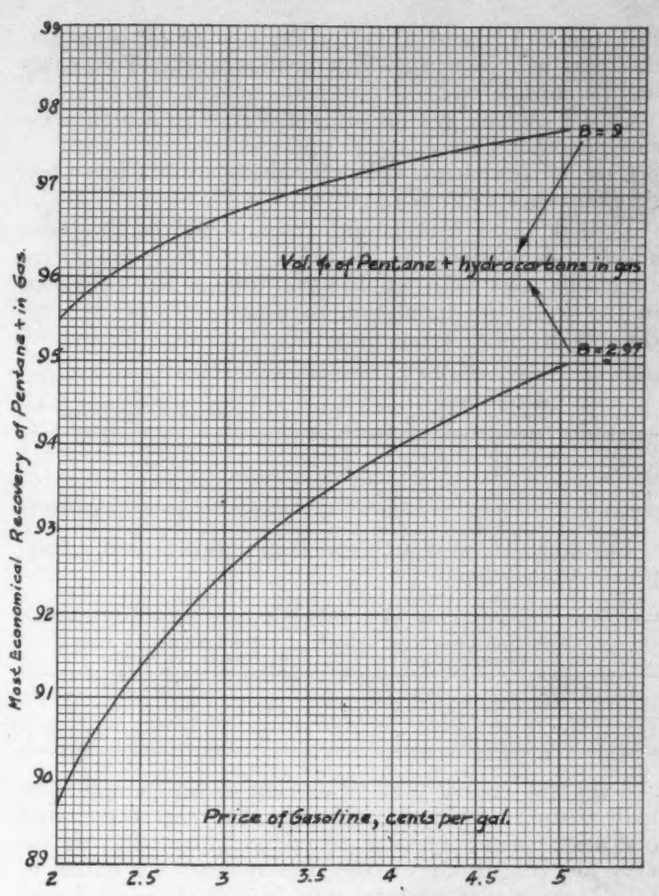


FIGURE 3

Most economical recovery of pentane when producing a 30-70 grade of Mid-Continent gasoline in a low pressure absorption plant. (50 pound.)

tended regardless of the quantity of material handled. Thus, the cost of oil recirculation amounts to little more than those extra maintenance and repair costs which are caused by the handling of additional oil and the cost of cooling water. Finally, some plants are using gas turbines by which power is produced. This even further reduces the cost of recirculating oil. The magnitude of the recirculating cost ranges from about 0.03 to 0.07 cents per gallon depending upon the size of the plant, the efficiency of the plant management, the value of fuel, and often most important of all, the cost of cooling water.

The above statements and low recirculation costs apply particularly to those isolated gasoline plants in distant oil or gas fields. They do not apply to absorption plants in refineries or in the vicinity of cities. In these plants the cost of power and steam is an important item and the cost of recirculation may amount to as much as 0.13 cents per gallon. With such a burden of recirculation cost, these plants cannot operate at a profit on lean gases and the economic recovery of pentane-plus seldom exceeds 93 percent.

The cost of stabilizing the raw natural gasoline is also low in the normal isolated gasoline plant for the same reasons that are listed above. However, there is an additional item, the loss in handling and storage, which is usually of great importance. The value of C_s is usually between 0.1 and 0.2 cents per gallon.

Inasmuch as plants are now usually operated for a pentane recovery of 99 percent, it will be useful to solve

Equations (12) and (13) for the cost of oil recirculation that will justify a 99 percent recovery. Two cases will be considered:

Case I. A lean gas (B = 3) absorbed in a high-pressure absorber (Eq. 13).

Case II. A rich gas (B = 9) absorbed in a low-pressure absorber (Eq. 12).

In both cases a 26-pound gasoline will be produced and C_s is considered to be 0.13. For Case I, the oil recirculation cost (C_o) would have to be as low as shown in the following tabulation to make a 99 percent recovery (R) most economical:

Gasoline Price C_g, Cents per Gallon	Oil Recirculation Cost C_o, Cents per Gallon Oil
3	0.0029
4	0.0039
5	0.005

For Case II, or a rich gas being absorbed in a low-pressure absorber:

Gasoline Price C_g, Cents per Gallon	Oil Recirculation Cost C_o, Cents per Gallon Oil
3	0.0089
4	0.0118
5	0.0149

Thus, the recovery of 99 percent of the pentane-plus hydrocarbons from a gas is not the most economical operation unless the cost of recirculating oil is of the very low magnitude of 0.01 or less cents per gallon of oil. Most plants operate at a higher circulation cost than this and hence the economic recovery of pentane will be less than 99 percent, and according to data which follow it will usually be economical to absorb less than 95 percent of the pentane-plus hydrocarbons.

EFFECT OF COST OF OIL RECIRCULATION

In general, the oil recirculation cost is the most important variable with which we deal. In order to further study the cost of oil recirculation, the most economical recoveries were computed for low-pressure absorber operating with an average or average-rich gas for the production of a 26-70 grade of Mid-Continent gasoline. The conditions are as follows:

Low-pressure, 50 lb. absolute.
Product, 26-70 grade gasoline.
Yield, 1.45 gal. for each gal. of pentane-plus recovered = y.
Pentane in gas, B = 6 percent.
Cost of stabilizing gasoline, C_s = 0.13 cents per gal.

These constants result in the following simplified equation:

$$R_E = 100 - 59\left(\frac{1}{C_g - .13}\right)^{.75}\left(C_o\right)^{.75} \qquad (14)$$

Using this equation, and allowing C_g and C_o to vary, the relation of recovery to circulation cost and gasoline price will be somewhat as shown in Figure 1.

LOW-PRESSURE ABSORBER

The most economical recovery for low-pressure absorbers are indicated in Figures 2 and 3. In both figures the following constants were used:

C_o = 0.05, cents per gal. oil recirculated.
C_s = 0.13, cents per gal. of gasoline stabilized.
K = 0.25, equilibrium constant for pentane-plus. (50 lb.)
M = 190, molecular weight of oil.
d = 0.84, specific gravity of oil.

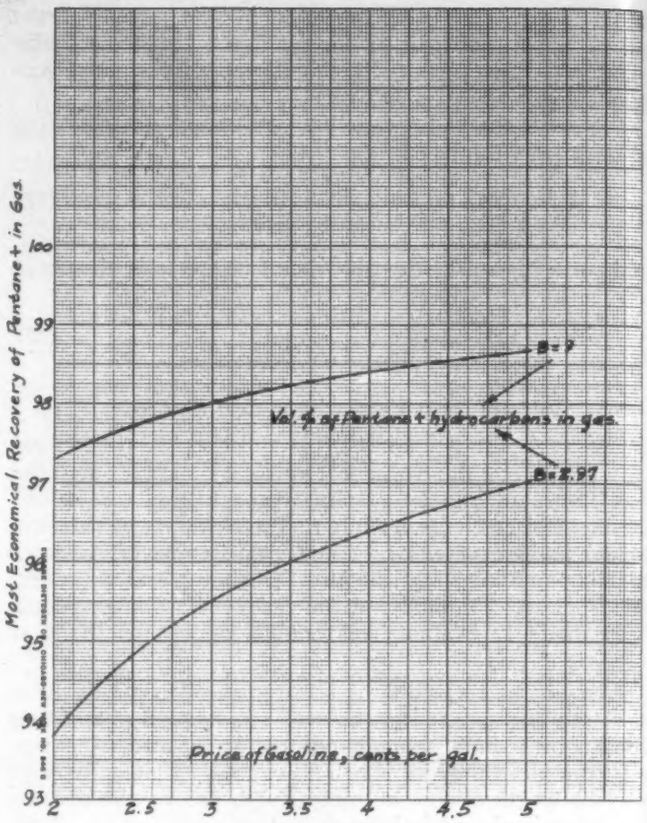


FIGURE 4

Most economical recovery of pentane when producing a 14-55 grade of Mid-Continent gasoline in a high pressure absorption plant. (400 pound.)

In addition 2 gases were considered—a lean gas containing 3 percent pentane-plus, and a rich gas containing 9 percent; and 2 gasolines were produced—a 14-55 grade, and a 30-70 grade.

Figure 2 shows the economical recovery when producing a 14-55 grade of gasoline from a rich and from a lean gas, and Figure 3 shows the same relationship but for the production of a 30-70 grade of gasoline.

HIGH-PRESSURE ABSORBER

The same general study was made for a high-pressure absorber and the results are shown graphically in Figures 4 and 5. The conditions were the same as for the low-pressure absorber studied above except that the pressure was 400 lb. per sq. in. absolute.

MAGNITUDE OF SAVINGS

The amount of money saved by operating at the economic recovery rather than at 99 percent recovery is of course the real test of the usefulness of the above formulations. It appears that if the cost of recirculating oil is small, then the saving in the total oil recirculation cost will also be small, and vice versa.

By returning to Equation (8) we may write:

$$P_R - P_{R_E} = 0.0037By(C_g - C_s)(R - R_E) - \left(\frac{2.38KMC_o}{3.156d}\right)\left(\frac{1}{(100-R)^{.33}} - \frac{1}{(100-R_E)^{.33}}\right) \qquad (15)$$

and for the case in which the actual recovery is 99 percent:

$$P_{99} - P_{R_E} = 0.0037By(C_g - C_s)(99 - R_E) - \left(\frac{.755KMC_o}{d}\right)\left(1 - \frac{1}{(100-R_E)^{.33}}\right) \qquad (16)$$

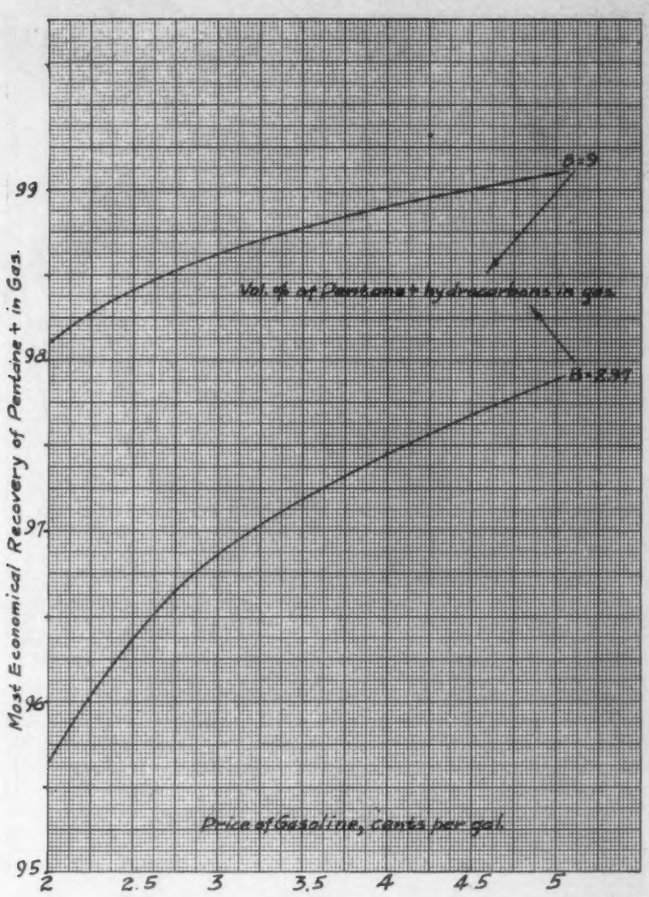


FIGURE 5

Most economical recovery of pentane when producing a 30-70 grade of Mid-Continent gasoline in a high pressure absorption plant. (400 pounds.)

Referring to Figure 2, which shows the production of a 14-55 grade gasoline from a lean gas, and considering a gasoline price of 4 cents a gallon—the economical recovery or R_E is found to be about 92 percent. Using this value and the corresponding constants in Equation (16)

$$P_{99} - P_{92} = 0.0037 \times 9 \times 1.07\ (4 - .13)\ (99 - 92) - \left(\frac{0.755 \times 0.25 \times 190 \times 0.05}{0.84}\right)\left(1 - \frac{1}{(100-92)}.33\right)$$

Loss by operating at 99 rather than 92 percent recovery = 0.965 — 1.42 = —0.455 cents per thousand.

This loss of about half a cent a thousand cubic feet amounts to about $5 a day for a million-cubic-foot plant. Although this is not a loss of great magnitude it is a loss that can be eliminated easily. It might be somewhat difficult to operate the plant for the exact recovery of 92 percent, but any recovery between about 76 and 99 percent will be more economical than operation at 99 percent recovery. A similar study of other operating conditions is given in Table 1. The cost of oil recirculation was 0.05 except in the case of refinery operating conditions.

TABLE 1

Loss by Operation at 99 percent Recovery

Pressure lb., per sq. in.	Pentane-plus percent	Grade of gasoline	Gasoline Price cents per gallon	Most economic recovery percent	Loss, cents per 1000 ft. by operating at 99 percent recovery rather than at R_E
50*	7	22—70	4	94.25	1.267*
50	3	30—70	2	89.8	0.821
50	3	30—70	4	94	0.577
50	3	14—55	4	92	0.455
50	9	14—55	4	96.2	0.286
50	9	30—70	4	97.4	0.227
400	3	14—55	4	96.4	0.117
400	3	30—70	4	97.5	0.068
400	9	30—70	4	98.9	nil

*Refinery conditions, $C_o = 0.1$, other conditions the same as other low-pressure plants.

Several generalizations can be seen in Table 1. The losses are greater at low gasoline prices, or when it is most necessary to make a profit. The losses in high-pressure plants are so small that operation at 99 percent recovery is satisfactory. The losses in refinery absorption plants are great because of the high cost of oil recirculation and operation at 93 to 96 percent recovery of pentane-plus is justified unless the plant is very small.

It has been shown heretofore that there is a particular recovery of pentane-plus which is the most economical recovery. As the recovery is decreased from 99 percent and approaches the most economical recovery, the profit increases. As the recovery is further reduced to values below the most economic recovery, the profit decreases again and finally is equal to the profit at 99 percent recovery. This low recovery which is economically as profitable as a 99 percent recovery can be determined by setting Equation (16) equal to zero, or

$$\frac{(99-R)\ (100-R)^{.33}}{(100-R)^{.33}-1} = \left(\frac{0.755KMC_o}{d}\right)\left(\frac{1}{0.0037By(C_E - C_a)}\right) \quad (17)$$

The equation must be solved by trial-and-error methods, but it could also be handled by plotting the

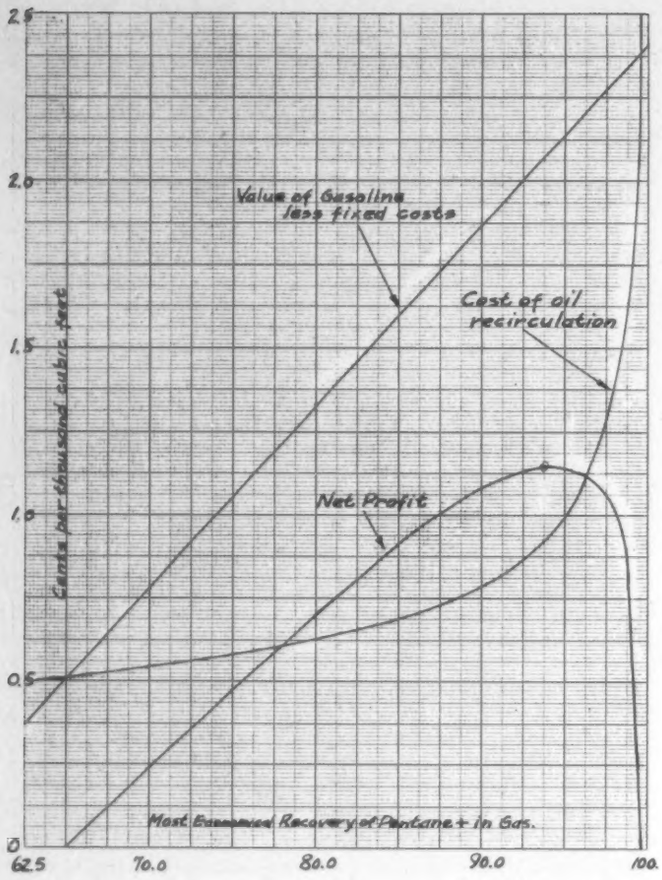


FIGURE 6
Net profit versus pentane recovery for low pressure plant.

"function of R" against R. Table 2 was prepared to illustrate the economical range of pentane recovery.

TABLE 2

Range of Pentane Recovery for Greatest Economy

Pressure lb., per sq., in.	Pentane-plus in gas, per cent	Grade of gasoline	Gasoline price, cents per gallon	Most economical recovery of Pentane	Range of economical recovery
50*	7	22—70	4	94.25	78 to 99*
50	3	30—70	4	94	81 to 99

*Refinery conditions, see Table 1.

Table 2 shows that it is more economical to operate at any recovery between 78 and 99 percent in a refinery absorption plant than at 99 percent recovery.

TABLE 3

Costs and Revenue for a Low-Pressure Plant

Recovery	Value of Gasoline cents per M cu. ft.	Recirculation costs	Gross Profit cents per M cu. ft.	Net Profit* (Gross less operation and depreciation) cents per M cu. ft.
60	3.24	0.498	2.74	—0.26 (loss)
70	3.78	0.547	3.23	0.23
80	4.33	0.625	3.7	0.7
90	4.87	0.786	4.08	1.08
93.72	5.065	0.922	4.143	1.143
95	5.135	0.995	4.14	1.14
99	5.35	1.7	3.65	0.65
99.5	5.38	2.14	3.24	0.24

*$C_d = 3$. This includes operation, supervision, supplies, taxes, depreciation and all costs that are not dependent on the recirculation of oil.

EFFECT OF NUMBER OF PLATES IN ABSORBER

The entire foregoing discussion has been limited to absorbers which contain 6 theoretical plates. In order to apply the method to columns having more or having less than 6 plates it will be necessary to derive equations similar to Equation (5) and insert this equation in the general form, Equation (8). The data from which to work, can be obtained from the absorption faction charts of Brown or Kremser.

In general, the most economical recovery of pentane-plus will be greater for columns having more plates than 6 and vice versa. Thus for absorbers containing only 4 plates the economical recovery will probably range from 82 to 97 percent whereas for 6-plate columns the recovery ranges from 90 to 98 percent.

SUMMARY

As a summary of the above results, Figure 6 and Table 3 show the profit when operating a low-pressure plant at the following conditions

$B = 6$	$C_s = 0.13$	$C_o = 0.05$
Grade = 26-70	$K = 0.22$	$C_d = 3$
$y = 1.45$	$M = 170$	
$C_s = 3.5$	$d = 0.83$	

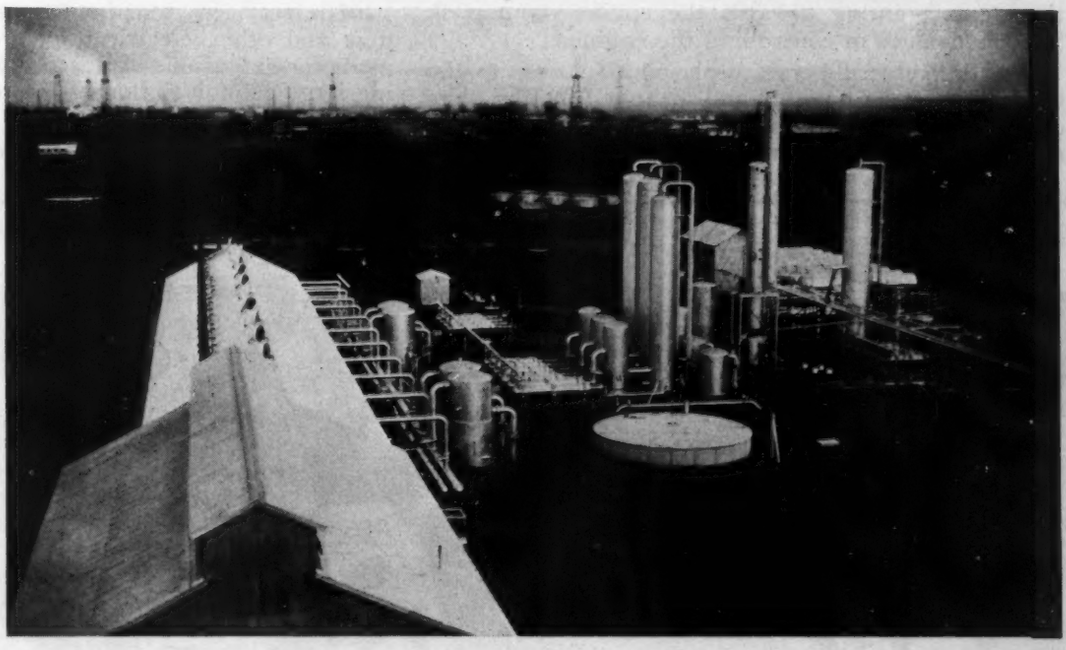
Thus for the particular conditions studied in Table 3 and Figure 6, the profit when operating at a recovery of 93.72 percent is almost twice the profit when operating at a recovery of 99 percent.

Design and Operation of

Fitts Gasoline Plant

J. C. ALBRIGHT

WHEN Carter Oil Company began laying out the ground plan for its Fitts gasoline plant, in the Fitts pool, Pontotoc County, Oklahoma, all of the gasoline plant experience was drawn upon so this plant would contain the operating advantages previously found satisfactory, and to eliminate the disadvantages uncovered in other plants when they had been operated for some time. The plant site was carefully selected in a large area controlled by the company, and the plant was laid out on a gentle southeast slope so all drainage would flow to a natural draw which runs through the property near the south boundary. The various units were placed in such positions that the cooling towers would occupy the highest point of the site. Boilers came next, being followed by the engine room, gas coolers, oil coolers, absorbers, distillation unit, fractionator, and finally the storage vessels were placed at the most distant point down the gentle grade. This was done so nothing of an inflammable nature could flow through the plant yard which might cause a serious fire.



Carter Oil Company's gasoline plant in the Fitts oil field, Pontotoc County, Oklahoma. Drainage is through gentle slope of plant yard to storage tanks shown behind fractionation column.

The field was comparatively young when the plant was erected, and the extent of drilling with subsequent production had not been determined. Adequate absorber capacity was placed in the plant to take care of several million cubic feet of gas, with provisions left in the design to add as the field grew. Eleven twin-cylinder, direct-connected gas engine driven compressors were installed in the engine room to handle the gas, since it must be taken from the traps and separators at a much lower pressure than the normal absorber operating pressure. These engines had already served in other plants operated by the company before they were moved to Fitts, but they were completely overhauled and rebuilt before being started here. The original design of the engines provided for the normal 165-horsepower delivery, with 16-inch power cylinders equipped with low-pressure fuel intake. New cylinders were purchased with a bore of 17 inches, and they were placed in an inverted position with the exhaust stacks extending through the roof of the building. Gas injection heads were provided which gave a rating to the overhauled engines of 230 horsepower each. New main and crank bearings were installed, as well as all other

needed parts which brought the condition of the units to that approximating new equipment.

One feature followed in this plant, which is not generally used by other gasoline manufacturers, is the manner in which the gas headers are arranged. Much difficulty had been encountered in other plants with corroded headers, which had been installed beneath the surface of the ground, and deteriorated because of soil conditions brought about by continual dampness. In the Fitts plant these headers both were installed well above the ground level and supported by concrete piers. Plugs used to close both ends of these headers were attached by flanges, which makes it easy for additions to be made. The pipes were treated at the point of contact with the piers to prevent localized corrosion, and the entire assemblies were then given a coat of aluminum paint.

The only part of the system between the compressors and the headers that is underground is the short pipes between the discharge stools on the compressors and where these pipes emerge from the ground outside the engine room and are flanged to the discharge header. Intake pipes take off directly from the suction header and rise to enter the building from an overhead point to be connected to the top of the compressor cylinders. All nipples used for both intake and discharge are welded directly to the headers on one end and to companion flanges at the others. Control gates are flanged to these nipples with the stems pointing towards the engine room walk for convenience in controlling the engines.

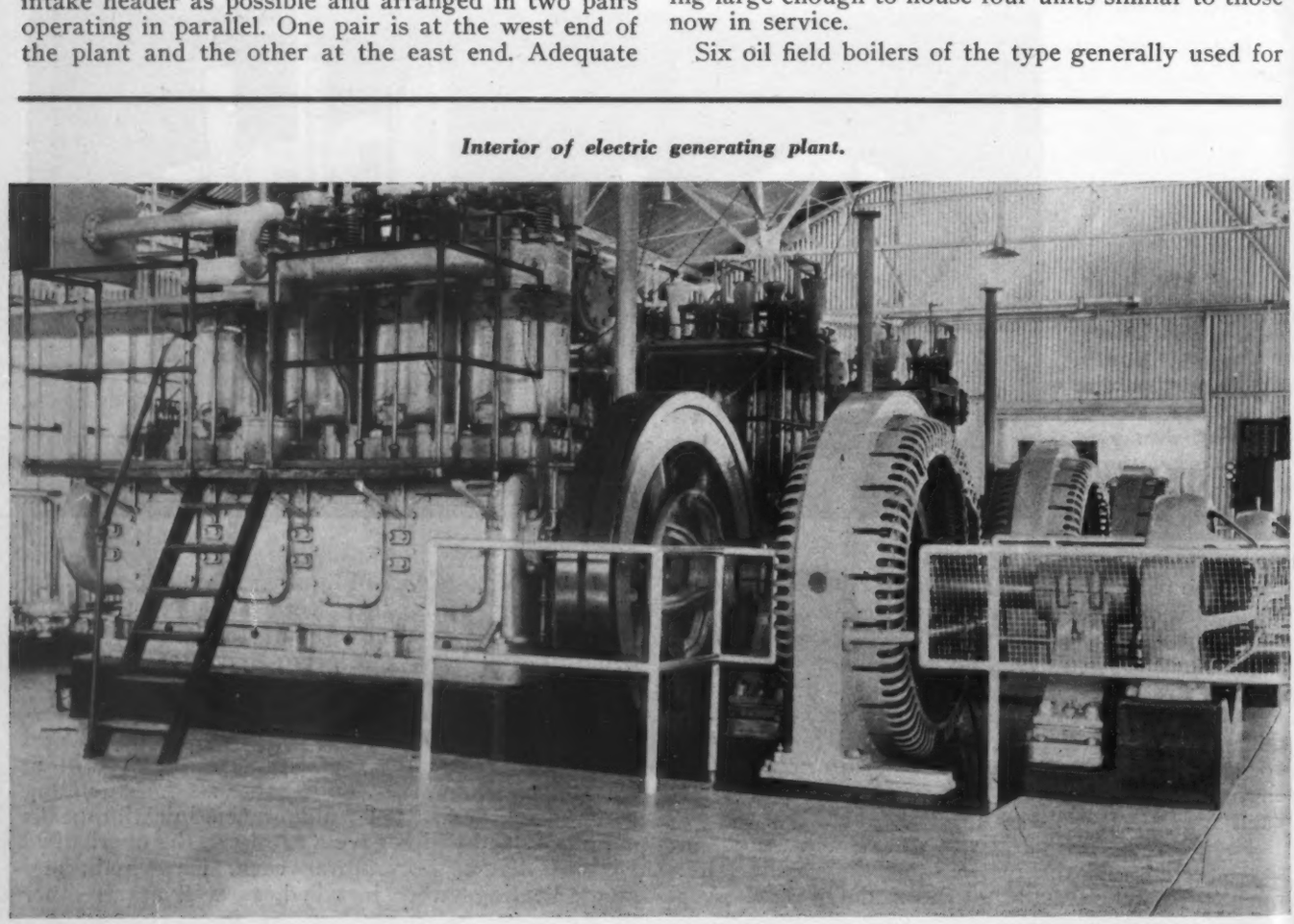
The intake scrubbers are placed as closely to the intake header as possible and arranged in two pairs operating in parallel. One pair is at the west end of the plant and the other at the east end. Adequate control gates are in lines leading to and from these scrubbers in order that any unit can be removed from service if the necessity arises. Drainage connections on all scrubbers permit removal of water and oil coming in from the field separators. Since the plant was started, in the fall of 1935, draining has been easily accomplished, because at some period of the day considerable back pressure is thrown upon the intake scrubbers by large volumes of gas being handled by the plant.

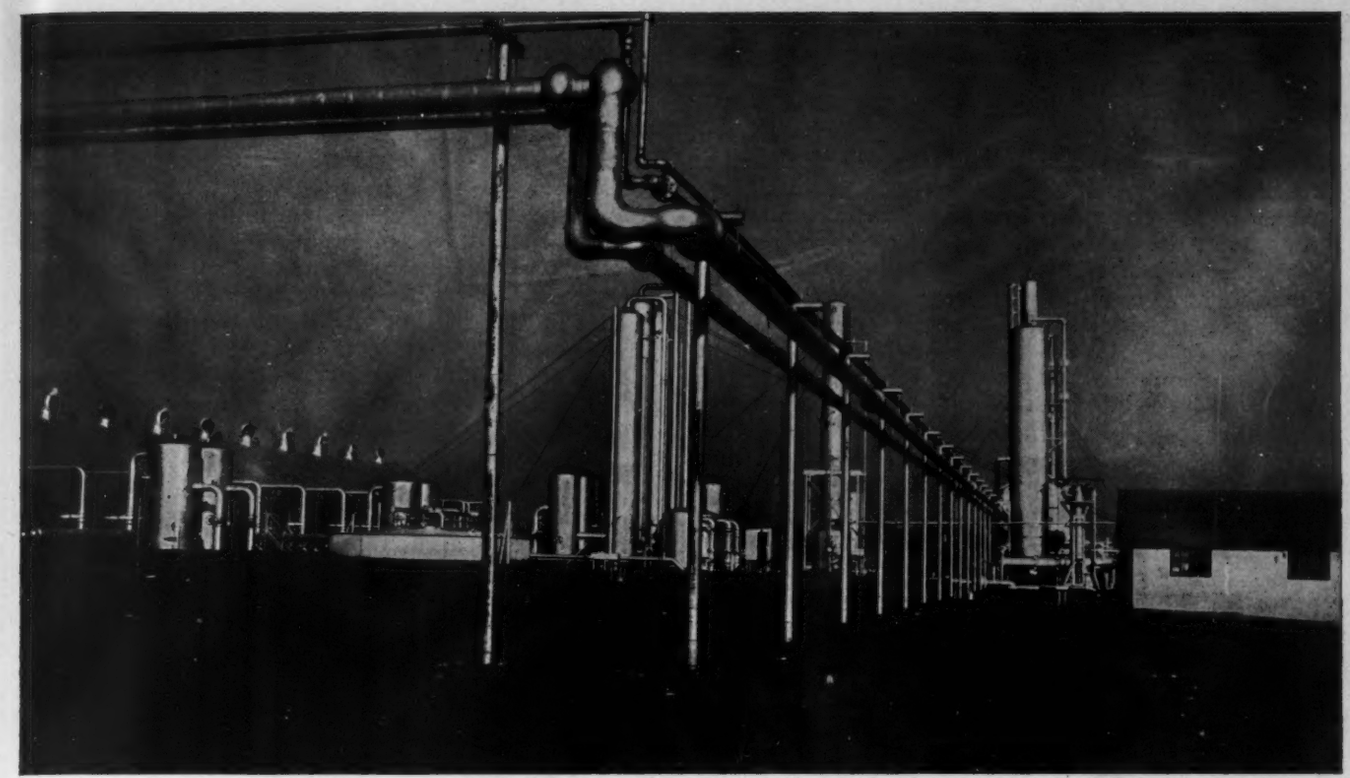
An auxiliary power room adjoins the compressor building on the end with a wide passageway between. This building is equipped with three units which generate electrical energy for plant operation and lighting. A large number of motor-driven pumps are in use in the Fitts plant and all power consumed by them is generated in the auxiliary power room. These units were moved from another location to be used in Fitts, but were in such good operating condition that it was not necessary to overhaul or rebuild them.

All power is transmitted to the various motor-driven pumps by underground cable, placed in this manner to eliminate erection of poles, which in many instances are obstructions to efficient operation. Junction boxes are placed at convenient points throughout the plant where branching lines are connected to the main cables. These boxes are built just above the ground, supported upon metallic legs, and covered. All plant lighting placed upon poles in the yard is serviced from below through standard conduit and other electrical fixtures. Provisions were made for extensions of this plant by erecting a building large enough to house four units similar to those now in service.

Six oil field boilers of the type generally used for

Interior of electric generating plant.





Steam and condensed steam lines shown supported upon steel posts with full swings at turn. Pipes hang below cross-arms from rods hinged in the cross-arms and to bands around the pipe lines which provides adequate movement for expansion and contraction.

low-pressure steam generation are located in a building adjoining the auxiliary power room on one end and the two cooling towers on the other. This boiler plant generates steam used for heating buildings, equipment exposed to the weather, and for steam used in preheating the oil and for process work generally. To reduce depreciation of the lines as much as possible due to soil corrosion, fuel and water lines are passed through the building walls and supported upon the steel trusses of the roof structure. The fuel lines take off from a common header placed on the wall just inside the building, gas being introduced into the header from a master regulator outside the boiler room on a setting above the ground level. The individual lead lines serving each boiler are extended from the header to the unit where they drop as risers to the burner connection on the right side of the firebox. They pass with the burner assembly through the brick foundation under the mud ring without coming in contact with the floor or the ground. Only one line is buried at the boiler room and that is the drain or sewer connection in front of the battery. It is made of large piping with sections removed from the top in front of each boiler. These sections are attached again to the pipe with hinges so the pipe can be closed between cleaning times for the boilers. The floor of the building is crushed stone, with the coarser material spread upon the sections of the drain so they can be opened easily. Individual drain lines lead from the sight indicators and the three hand cocks to drain dripping water to the sewer without marring the front of the boilers and damaging the insulation.

Conventional type steam-driven water pumps are used as boiler feed units, taking water from a tank outside the building and supported upon steel legs. This tank serves the purpose of a feed-water tank and for a receptacle where condensed steam is returned from the preheaters, reboilers and distillation units in the plant. Make-up water is pumped as needed from a closed steel tank placed on concrete cradles at the end of the building, and is always kept at a predetermined level through automatic controls operating the make-up feed water pump. The condensate from the plant was fed to the aerial tank through a perforated line when the plant was first built, but afterwards was removed because of the rapid condensation of steam which generated a troublesome water hammer in the lines. Blow-down lines lead from the back of the mud ring through two lubricated plug valves to a common header placed just outside the building. Each line is connected to this header by welding a tangential nipple to the side of the header to reduce the impact of the blow-down and to induce a whirling of the water and steam as a primary muffler or noise preventer. In the center of this header a large vertical stack extends well above the eaves of the building which dissipates the uncondensed steam in the air so it will not come in contact with other equipment. A large drain connected to the base of the standpipe in turn is attached to the boiler room sewer line so the blow-down drains from the plant with the rest of the sewage. The header is made of 10-inch pipe and the large standpipe built from salvaged tankage with a diameter of about 3 feet. It is so efficient as a noise reducer that when standing close to the boiler room, the blow-down is not noticeable.

All lines, whether carrying steam under boiler pressure or returning condensed steam from the

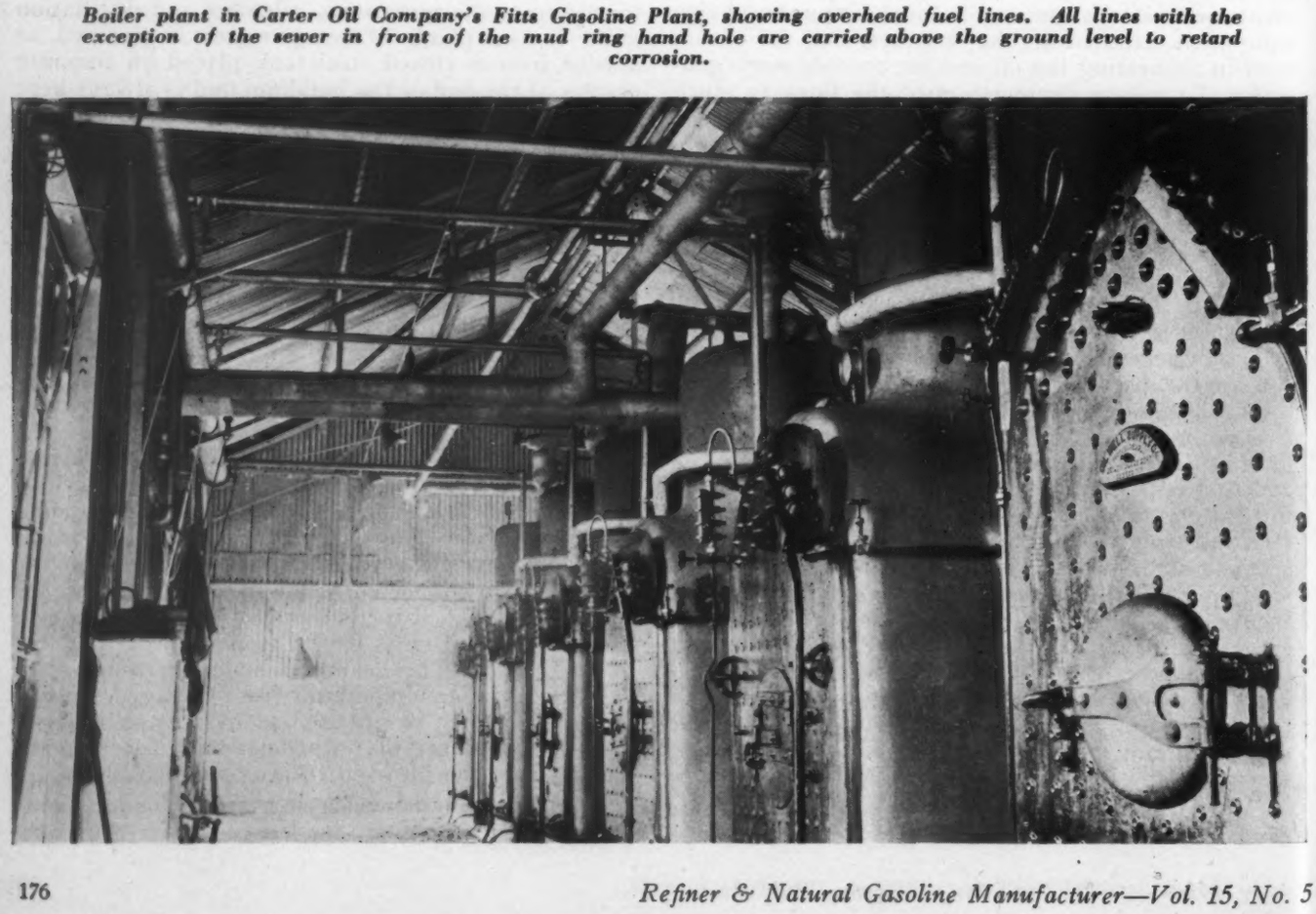
plant at the opposite end of the yard, are carried above the surface of the ground. Steel pipes were used as supporting poles with cross-arms welded to the top of each. These cross-arms were drilled through the upper side and long slots were cut through the lower. Long eye-bolts were placed in the slots and extended through the holes in the upper side. The bolts were furnished with threads on the opposite end from the eye, upon which nuts were screwed after being placed in position. The steam lines were fitted with straps at each post and attached with bolts. Each end of the straps was bored to receive the bolts, and when installed resembled clamps on the pipe. As the pipe was made up, the bolts tying the straps together were passed through the eye in the hanger bolts. Complete right-angle turns were made in carrying the steam lines through the plant yard, and when the turns were necessary, the supporting pipes dropped in length a sufficient amount to accommodate long, full-swing joints. As the steam lines were necessarily of considerable length, a large amount of expansion and contraction was expected. Coupled with the vertical hanger bolts, a wide latitude of movement is permitted by virtue of the slotted holes in the cross-arms, and the long full-swing connections in the pipes themselves, and no stresses are set up which would damage the steam lines or connections. Steam lines as well as all other lines effecting boiler operation are carried on these supports, the lowest of which has a sufficient height that trucks may pass at any point without interference.

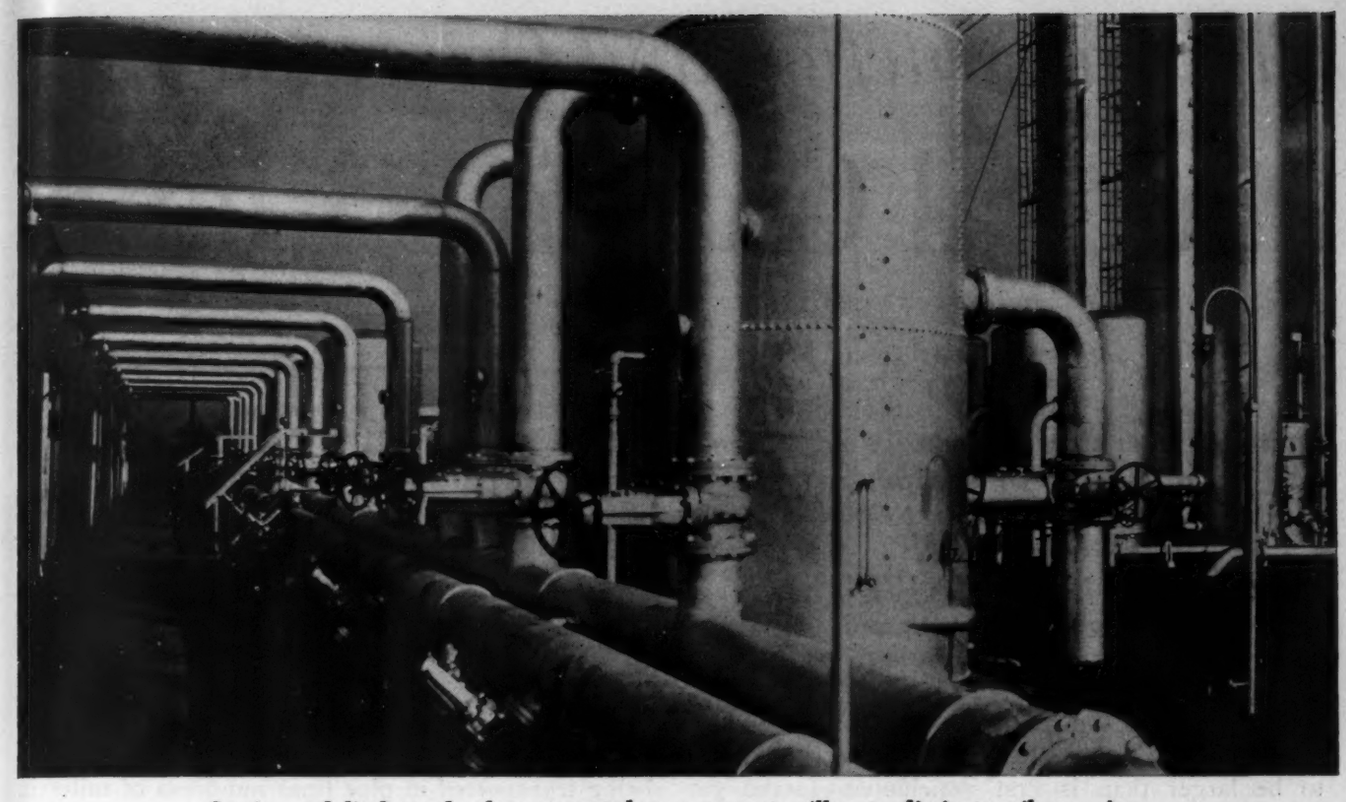
Water for plant consumption and cooling is obtained from a field system. Enclosed shell and tube units are used in the plant for gas and oil cooling, but engine jacket water is carried through a separate system. Atmospheric sections were placed in one of the two cooling towers through which jacket water is pumped for cooling. The water originally placed in this system was not softened, and no harmful effects have been discovered from scale deposition, Make-up water for this system is taken from a supply condensed in the oil preheaters in the plant. The water is picked up by centrifugal pumps, after it flows over the engines by gravity from a small tank, from a half-buried hot well down the slope from the engine room. Not all of the water used in the plant is run through the softening unit, as it is not desired to maintain the circulation at zero hardness.

The gas cooling units, which are closed shell-and-tube equipment, were placed directly between the compressors and the absorbers. During the early spring months, the gas enters the absorbers at a temperature of approximately 60° F., which is only about 5° F. above the temperature of the water leaving the cooling tower basins. After the gas has passed through scrubbers between the cooling sections and the absorbers, and through after-scrubbers to remove any trace of mineral seal oil, it is passed to the field for fuel in drilling. Part of it, however, is picked up by other engines and boosted to the pressure necessary to complete deep wells when drilled in "alive." This pressure varies according to the formation in which the oil is found, and may run as high as 700 pounds at times.

Three absorbers are used in this plant with each column taking its share of the gas for stripping. Provisions were made when laying out the plant so

Boiler plant in Carter Oil Company's Fitts Gasoline Plant, showing overhead fuel lines. All lines with the exception of the sewer in front of the mud ring hand hole are carried above the ground level to retard corrosion.





Suction and discharge headers supported upon concrete pillars to eliminate soil corrosion.

that additional columns can be erected and placed in service without disturbing operation of those already installed.

Rich and lean oil is pumped through the plant with electric motor-driven centrifugal pumps which are set in the open as near the units served as possible without crowding. The motors were made explosion- and water-proof which makes it possible to operate them without housing. The lean oil, with a gravity of 41.8° A. P. I., enters the absorbers at a temperature of about 63° F., but the heat of absorption increases the temperature to about 74° F. After passing through the first exchangers and entering the rich oil vent tank, the temperature has been increased to about 158° F., but the preheater increases this to about 250° F., and it is processed in the still at 325° F. The pressure maintained upon the still averages about 40 pounds gauge ,with process steam being introduced from a line with 125 pounds pressure. The distillation column is a single unit which is maintained with a top temperature at the knock-out box of 184° F. to prevent carry-over of mineral seal oil. Vapors from this unit are passed through shell-and-tube condensers and liquid leaves them at 60° F. Vapors from the still which are not condensed to a liquid in the condensers are carried through a tail absorber which extracts the desired fractions upon contact with a stream of mineral seal oil circulating through the system with that from the main absorbers.

As much as 22,000 gallons of finished gasoline per day has been manufactured in this plant, but the volume varies with the production methods followed in the field. During peak periods, as much as 34,000,000 cubic feet of gas has been handled by the 11 compressors. The varying amount of gas available for processing, running from low to high volumes, makes for an aggravating operating condition. When large volumes are available, the pressure sometimes jumps to as much as 10 pounds back pressure on the intake of the engines which crowds out the richer gas coming from the wells being pumped or flowed under low pressures on the gas outlet of the traps and separators.

Gasoline, upon being accumulated in the distillation plant receiving tank, is pumped to the fractionator where it is converted to the desired vapor pressure. Normally the plant produces the usual type of product, being classed as a 26/70 natural. The fractionating equipment was made especially for the Fitts plant and the column is 36 inches inside diameter by 60 feet in height. As the gasoline passes from the base of the column, it is directed through a treating unit placed in a building near the unit. This plant uses a concentrated chlorinated lime as the treating agent.

The gasoline is shipped in tank cars which are loaded at the nearest railroad station. The loading line passes over some very rough terrain and parts of it are almost inaccessible except on foot. The finished product is pumped into the line with a piston type pump driven by a 20-horsepower electric motor, and the line is kept under constant pressure at all times to prevent vaporization of the gasoline. While the loading pumps are idle, the line is protected by a "theft detector" designed by Carter Oil Company engineers and which sets an alarm signal to sounding when any sudden drop in pressure occurs at any point on the line. This apparatus operates on a drop in pressure across a small orifice plate which in turn sets in operation a mercury switch connected to the plant signal system. It is said that only a very small amount of gasoline need be removed from the line to set the theft detector ringing.

The Construction and Operation of
Natural Gasoline Plants

PART II

Factors governing the choice of process and types of equipment

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IN a recent publication of this magazine[1] various economic factors were discussed which have an influence upon the location of a plant site for a natural gasoline plant. In this paper, the same subject is given further consideration as well as the matter of the selection of the most suitable manufacturing process.

PROVISION should be made for expansion in the size of the plant in the event that the field proves to be larger than is first anticipated. Intake gas scrubbers usually are placed at some distance from each end of the compressor room in order to obviate the necessity of moving the scrubbers if more compressor units are added. Likewise the absorbers, distillation units, boiler house, cooling tower, storage tanks, etc., should be so placed that expansion can be made without interference. This principle can best be shown by a typical plant layout, as given in Figure 1. By reference to this drawing, it is easily seen that expansion of any one of the various units making up the plant can be made in a longitudinal direction. This rule applies of course primarily to those locations where fairly level land is available in all directions.

WATER SUPPLY

Gasoline plants are dependent upon a steady supply of water for successful operation. Everything else being equal, the plant site nearer the water supply is better, in that water pumping and transportation costs may be reduced. Since gasoline extraction of any kind is essentially one in which the latent heat of condensation, of the heavier fractions in the gas, is removed, it is necessary to cool the large quantities of water which absorb this heat. The presence of scale-forming constituents in the plant water is undesirable as they add greatly to the maintenance and operation costs. If a plant is located near a river, lake or sea, investment in cooling towers may not be necessary if the water source is as cool as the water temperature attainable by cooling tower operation, and the cost of delivering the hot water a sufficient distance away is not excessive.

RAILROAD AND PIPE LINE FACILITIES

Proximity of a railroad to a plant is desirable not only from the standpoint of lowering shipping costs of the natural gasoline and other products but also because supplies such as plant equipment, absorption oil, lubricating oil, etc., can be obtained more cheaply. Large quantities of natural gasoline are being transported in pipe lines hundreds of miles in length with but small losses, and at much lower costs than by rail shipment. The blending of natural gasoline directly with crude oil in the pipe lines is sometimes employed, if the mixture of crude oil and gasoline is being pumped straight to a refinery. If long storage periods are expected, such practice is not advisable on account of evaporation losses. Natural gasoline served the purpose of reducing the viscosity and lowering the pour test of crude oil which was pumped from the Borger field in the Texas Panhandle between 1927 and 1930. Ten percent of natural gasoline by volume in this crude oil mixture resulted in an increase of 25 percent or more in the crude oil deliveries to the Gulf of Mexico.

TOWNSITE LOCATIONS

There are certain advantages in having a gasoline plant fairly close to some town. The necessity of erecting dwellings for the employes is not so great. Less travel is required for employes in order to do their marketing. Better school facilities generally are available for the children. At the same time it is well not to locate a plant so close to a town or city that future growth of the town may result in the inclusion of the plant within the city limits. Taxes are invariably higher within a city and the danger of lawsuits resulting from a fire is apt to be greater due to the more probable loss of life and property.

DAMAGE FROM PLANT DRAINAGE

The drainage of waste oil and spent chemicals away from the plant is desirable, but that alone does not necessarily solve the problem of disposing of plant refuse. To prevent waste, such as crude oil, or spent chemicals, as calcium chloride, from causing damage to nearby streams or crops is a big problem. Oil can be collected easily and used as fuel or otherwise be burned in open pits. Earthen sumps for the collection of oily waste should be so located that sudden rainstorms will not cause them to overflow

and contaminate streams. The disposal of salt waste is by far more difficult. The most satisfactory method is that of storing the salty solutions in dry weather and releasing the waste into nearby streams during flood stage. If the volume of salt water is large, evaporation by artificial means is often resorted to in order to reduce the size of the storage reservoirs required to hold the solution from one rainy season to another.[4] The ever increasing number of damage suits based on stream pollution attests the importance of serious consideration of the waste disposal problem.

TYPE AND SIZE OF PLANT UNITS

In practically all new fields the gasoline content of the gas runs less than two gallons per thousand cubic feet. The oil-absorption process usually proves to be the best one for handling these flush gas volumes. The compression process is satisfactory for rich gas containing 5 gallons or more per 1000 standard cubic feet.

Between 1925 and 1930 the charcoal process was given a thorough trial in low-pressure plants throughout the Mid-Continent area. Some of these plants proved to be fairly successful when located in areas where gas was available in large quantities and also under 30 to 50 pounds back-pressure. The greatest difficulty with the operation of charcoal plants was due to the pulverization of the charcoal whenever the linear velocity through the adsorbers proved to be excessive. Maintenance of screens holding the charcoal in place also proved to be quite costly.

Crude oil mist had to be removed from the incoming gas in order to maintain the activity of the charcoal. This removal was accomplished either by glass wool scrubbers or charcoal pre-absorbers. The colloidal nature of the mist called for a neutralization of the electrical charge on the crude oil particles by some surface having an opposite charge such as glass or charcoal. Mechanical precipitation of such mists proved ineffective.

Another type of charcoal contamination came to those plants operating in sour gas areas. Whenever the sulphur-bearing gases were brought into the plants under vacuum, the infiltration of only a frac-

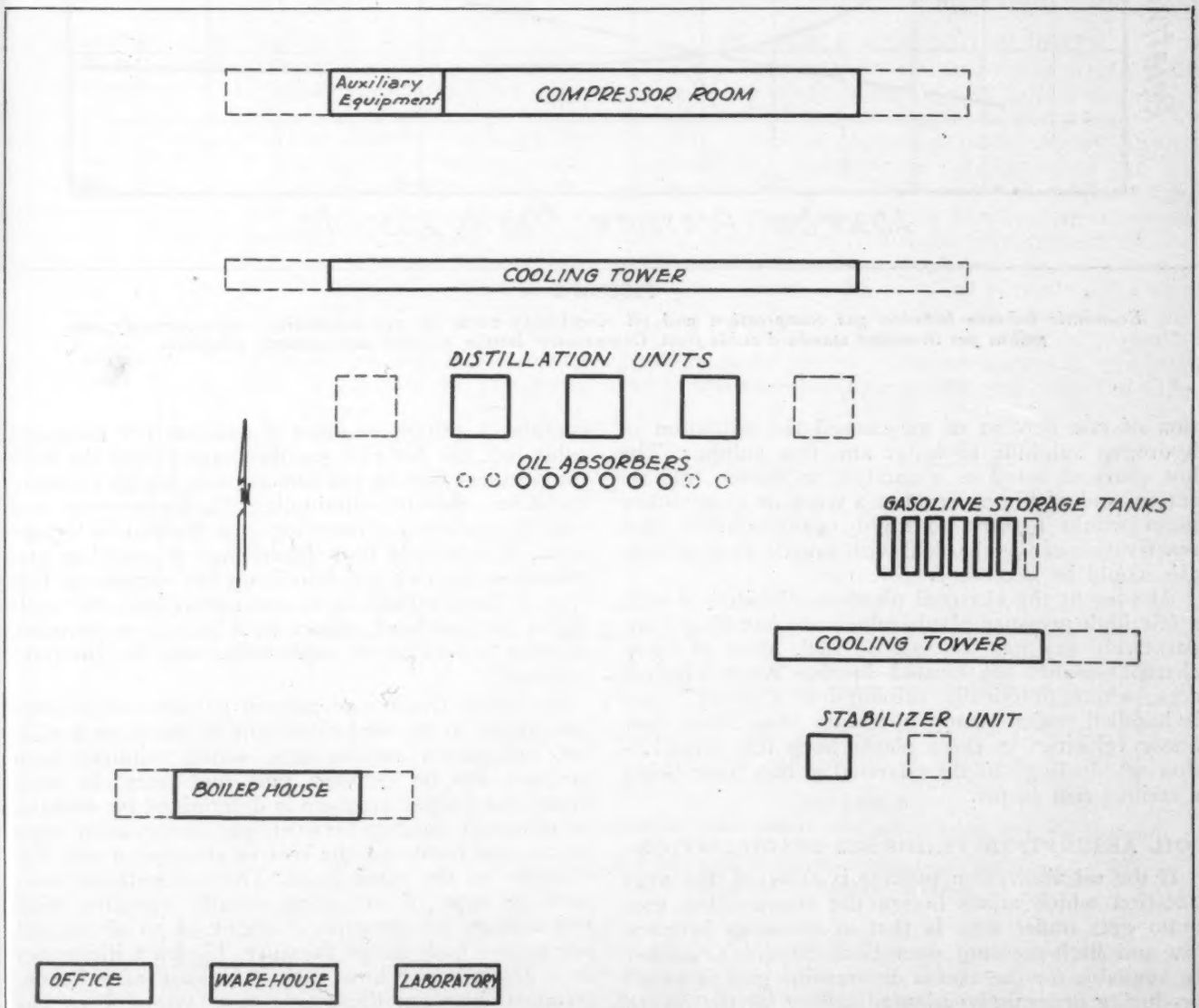

FIGURE 1
Typical Gasoline Plant Layout

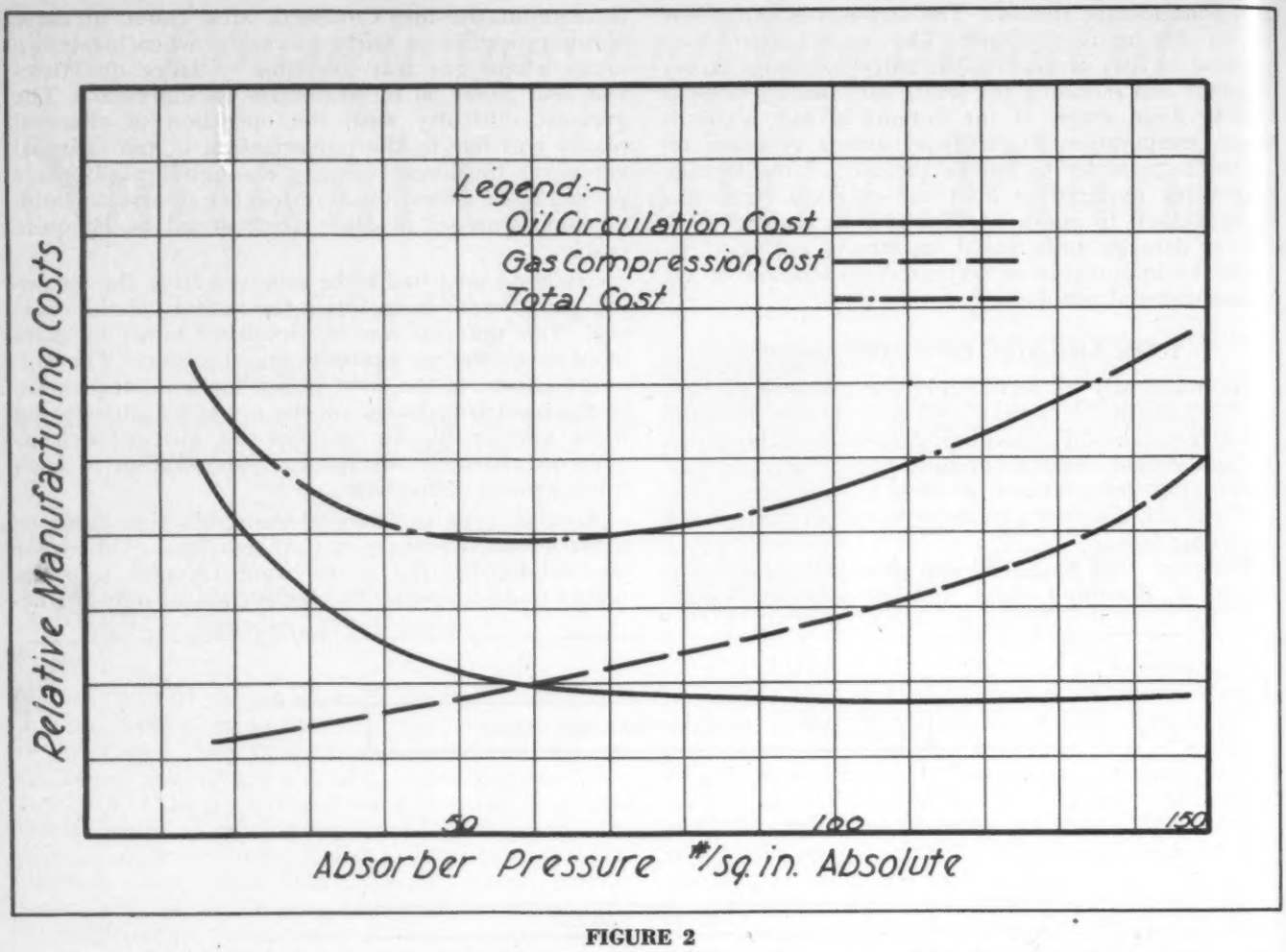

FIGURE 2

Economic balance between gas compression and oil circulation costs on gas containing approximately one gallon per thousand standard cubic feet. Compressor intake around atmospheric pressure.

tion of one percent of air caused the oxidation of hydrogen sulphide to water and free sulphur. The hot charcoal acted as a catalyst to hasten this reaction, and within a period of a week or so an entire plant would become so highly contaminated that reactivation of the charcoal with caustic soda or soda ash would be necessary.

At present the charcoal plants are limited to only a few high pressure plants which are handling comparatively dry gas for sale as fuel. Most of these charcoal plants are located in the West Virginia area, where practically sulphur-free ("sweet") gas is handled under steady operating conditions. Low linear velocities in these plants keep the pulverization or "dusting" of the charcoal grains from being a serious cost factor.

OIL ABSORPTION VS. DIRECT STABILIZATION

If the oil absorption process is selected, the next question which arises before the construction program gets under way is that of choosing between low-and high-pressure operation. Should a market be available for the excess dry residue gas, or an oil producing program be adopted calling for the return of excess residue to the oil sand for repressuring or gas conservation, the logical choice is a high-pressure oil-absorption plant. If, however, the gas contains 5 gallons or more of gasoline per thousand cubic feet, the hot rich gas discharged from the high compressors may be put directly into a high-pressure stabilizer, thereby eliminating the investment and cost of operating absorption and distillation equipment. A relatively high percentage of methane and ethane in the rich gas handicaps the success of this type of direct stabilization, and necessitates the cooling of the overhead vapors with butane or propane, in order to provide an ample reflux ratio for the fractionator.[1]

The large fraction of natural gasoline oil absorption plants in the Mid-Continent do not have a market for excess residue gas which requires high pressure for its delivery into fuel lines. In such plants the proper pressure is determined by striking an economic balance between gas compression costs on the one hand and the cost of absorption and distillation on the other hand. The conventional low-pressure type of oil plant usually operates with single-stage compression at about 35 to 40 pounds per square inch gauge pressure. Figure 2 illustrates in a general way how such a balance may be determined between these two cost factors, i. e., gas compression and oil circulation.

The rapid decline of gas volumes in new fields producing up to their capacity causes the average

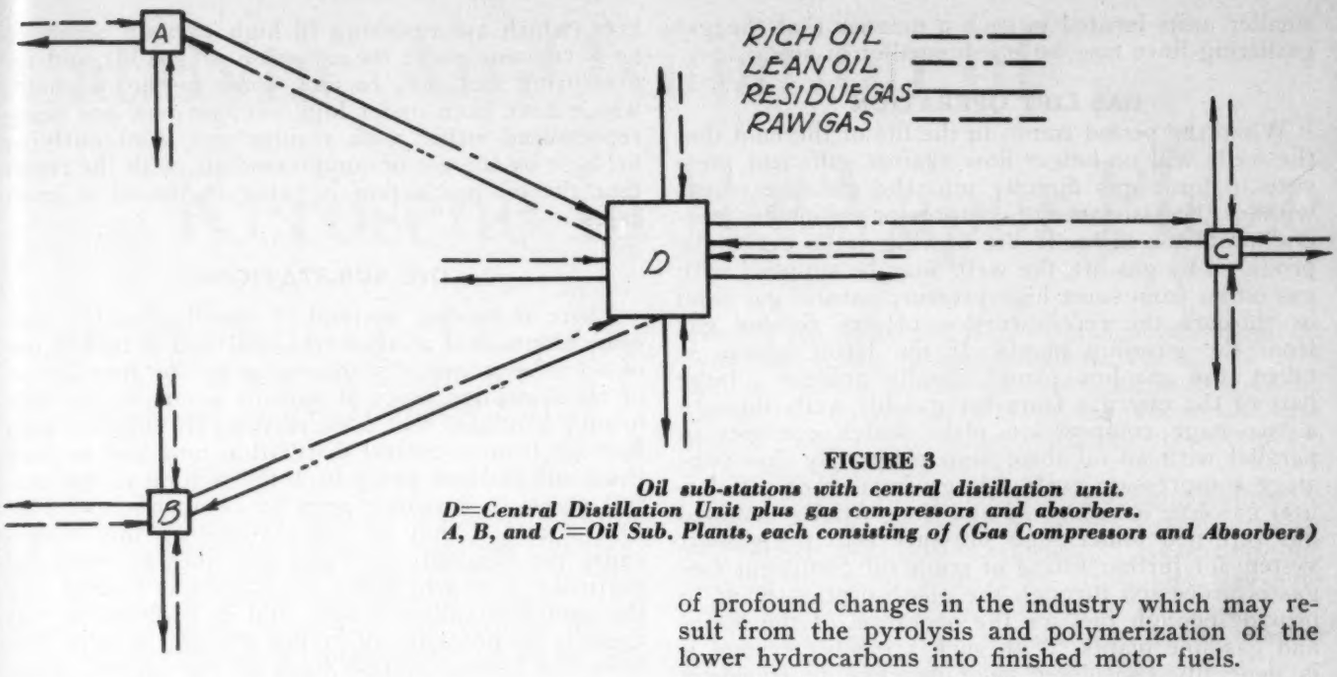

FIGURE 3

Oil sub-stations with central distillation unit.
D=Central Distillation Unit plus gas compressors and absorbers.
A, B, and C=Oil Sub. Plants, each consisting of (Gas Compressors and Absorbers)

gasoline plant to be short-lived. When a new plant is proposed the first question which arises is whether the equipment is to be transferred from an old plant, or should more recently designed units be installed to take the place of the less efficient obsolete plant which may still be in fairly good operating condition. This factor of obsolescence exacted a big toll on many relatively new plants during the decade 1920 to 1930 when kaleidoscopic changes occurred in the methods of extracting gasoline and in fractionating the raw gasoline product. Although research at present points toward the development of new processes and products, it appears to be improbable that such a rapid revolution will occur during the present decade. There is of course the possibility of profound changes in the industry which may result from the pyrolysis and polymerization of the lower hydrocarbons into finished motor fuels.

PORTABLE ABSORPTION UNITS

If the producing wells can flow under a high back-pressure, the usual custom is to install an absorption and distillation unit somewhere in the proximity of the first group of producing wells. About 1925, it appeared that portable-type oil-absorption and distillation units might solve the problem of harnessing large flush gas volumes. This development did represent a forward step for those fields where the gas could be obtained under sufficient natural flowing pressures. Unless these so-called portable units were fairly large in size, however, the operating cost proved to be exorbitant. As a result, the smaller portable units proved to be unsuccessful, and only the larger semi-portable plants survived. Proration

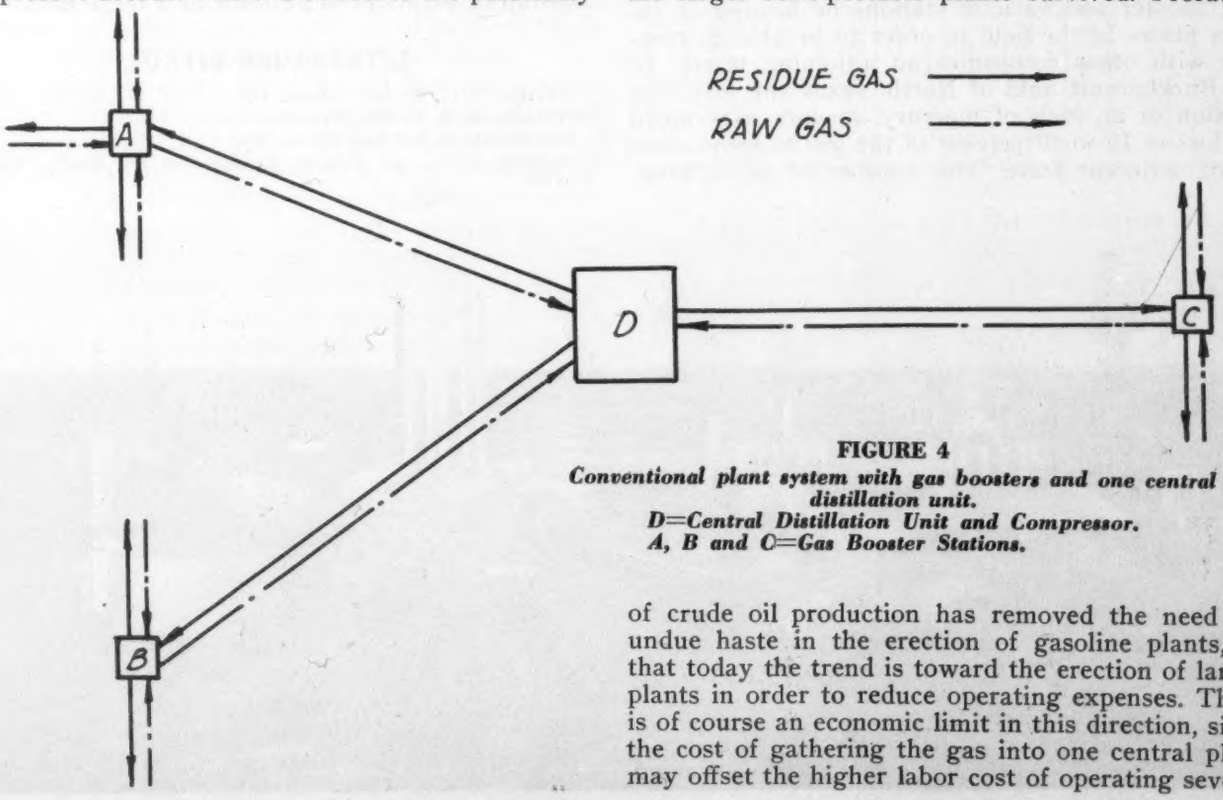

FIGURE 4

Conventional plant system with gas boosters and one central distillation unit.
D=Central Distillation Unit and Compressor.
A, B and C=Gas Booster Stations.

of crude oil production has removed the need for undue haste in the erection of gasoline plants, so that today the trend is toward the erection of larger plants in order to reduce operating expenses. There is of course an economic limit in this direction, since the cost of gathering the gas into one central plant may offset the higher labor cost of operating several

smaller units located in such a manner that the gas gathering lines may be much smaller in size.

GAS LIFT OPERATION

When the period comes in the life of the field that the wells will no longer flow against sufficient pressure to force gas directly into the gasoline plants without the assistance of compressor equipment, several problems arise. If the flowing wells are to be produced by gas lift, the wells may be supplied with gas either from some high-pressure natural gas sand or through the recompression of dry residue gas from the gasoline plants. If the latter course is taken, the gasoline plants usually process a large part of the raw gas from the gas lift wells through a two-stage compression plant which operates in parallel with an oil-absorption plant.[2] By this two-stage compression cycle a large fraction of the natural gasoline is recovered and the partially denuded gas returned under high pressure into the gas-lift system for further lifting of crude oil. Sufficient raw gas is processed through the oil-absorption plant to provide enough fuel for the operation of the leases and gasoline plants. With such a gas-lift system it is generally considered poor practice to transport gas more than three or four miles because of the excessive cost of pipe lines.

When several plants are installed by one company in a large producing field it is customary to connect these plants together with both raw and residue gas lines, so that in the event one plant is partially dismantled or abandoned the gas can be handled through the remaining plants. Then, too, sometimes the oil-absorption and distillation equipment of one plant can be later abandoned, while the compressor equipment may be used to boost the gas to one of the other plants which may have sufficient absorption and distillation capacity to handle more gas. In some of the older fields where high vacuum is being pulled on the oil-producing sands it is quite essential that booster and vacuum stations be located at various places in the field in order to be able to compete with other companies on adjoining leases. In the Burkburnett field of North Texas the loss of a fraction of an inch of mercury vacuum may mean the loss of 10 to 20 percent of the gas to some competing adjacent lease. The number of fields, however, which are resorting to high vacuum seems to be decreasing since the adoption of gas-lift and repressuring methods. In fact, some of the old fields which have been under high vacuum now are being repressured either with residue gas from outlying fields or by the use of compressed air, with the result that the oil production is being increased in most cases.

OIL SUB-STATIONS

There is another method of distributing the gasoline plants over a large field, and that is by the use of oil sub-stations. By this is meant the installation of oil-absorption units at various points in the field in such a manner that these stations are supplied with lean oil from a central distillation unit and in turn these sub-stations pump back the rich oil to the central plant. It is readily seen by comparison of Figures 3 and 4 that oil sub-stations of this nature cause the elimination of raw gas lines between the particular area where the sub-station is located and the central distillation unit, and in addition to this there is no necessity of laying a return residue line from the central station back to the oil sub-plant, since the dry residue gas is produced at the sub-plant itself. Offsetting the raw and dry gas lines, however, is the cost of lean-and-rich oil lines and also the oil pumping equipment. Although this method of distribution has proved satisfactory in certain cases, its adoption has not been general. This is probably due to the fact that the operation is more or less complicated by pumping large quantities of absorption oil back and forth over the field. The loss of absorption oil from line breakage is apt to add greatly to operation costs. The oil sub-station may still come in for a certain amount of play in the future, but from all indications its use will be more or less limited. To say the least, the economics of such an installation should be considered whenever a group of plants is to be built in a large field.

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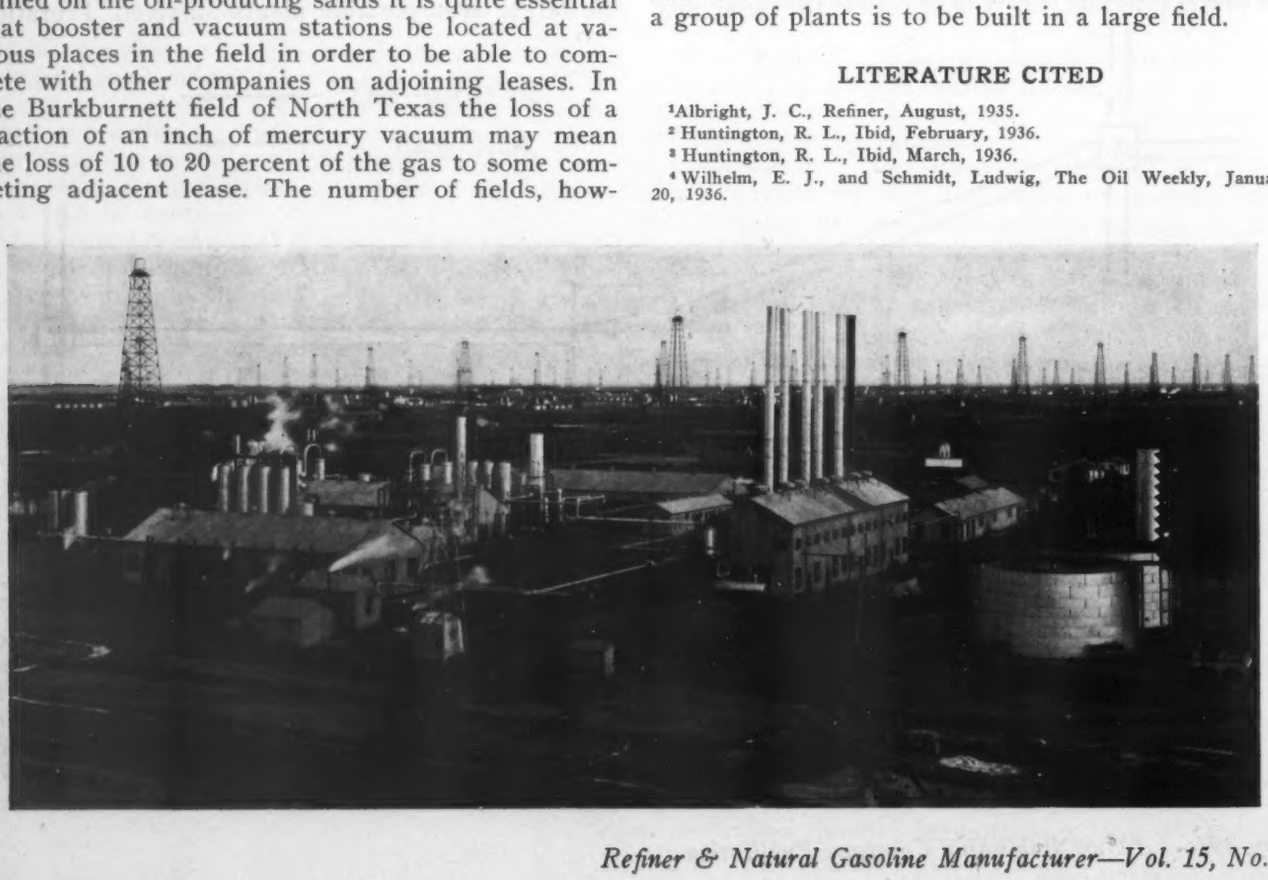


Catalytic Air-Oxidation of Kerosene in the Vapor-Phase

DR. RUDOLF KLATT and DR. HANS JELINEK
Translated from PETROLEUM ZEIT.

THE object and purpose of the work before us was to determine the best oxidation conditions for converting a naphthene-base Russian kerosene into the greatest possible quantity of oxidized water-soluble compounds, as well as to determine the nature of the oxidation products formed under our particular conditions.

The apparatus used is described in Petroleum, Vol. 30, No. 13, page 6 (1934), except that at present the reaction tube employed is made of galvanized iron, and the flasks, in which the vaporization of the oil and water take place have now been constructed of copper. The connection between the flask and the tube is made by means of a union-joint.

Five percent of vanadic acid deposited on 95 percent of granular pumice-stone (kernels) is the oxidation catalyst employed.

The determination of the optimum oxidation conditions for the kerosene in question was arrived at by means of a series of preliminary tests. These were carried out so that always 50 cc. of oil could be simultaneously vaporized with 150 cc. of water. Fifty cc. of water was always carried in the 10-bulb tube and 100 cc. of water in the Wulff flask.

All the preliminary tests showed that the reaction product consists of a water-soluble yellow-brown colored portion of highly acidic nature; it has a very pungent odor; besides that, a water-insoluble fraction is obtained also, which is lighter in color, and lighter in gravity; finally, one also notes that at the interface of these two portions, there has accumulated a whitish voluminous precipitate.

In order to characterize the various runs, the products formed in the reaction were tested as follows:

1. The acids which had formed were determined by the titration of 50 cc. of the watery portion with N/2 sodium hydroxide (phenolphthalein). Multiplying the number of cc. of alkali consumed by 6—for the basis of 300 cc. of the watery portion—one obtains the amount of N/2 acids formed per cubic centimeter.

2. The aldehydes formed are determined as follows by the method of Blank and Finkenbeiner[1]: 50 cc. of the watery portion are neutralized with N/2 sodium hydroxide (using phenolphthalein) and then treated with 25 cc. of a 4 percent solution of hydrogen peroxide (neutral to the same indicator), and 50 cc. of a N/2 sodium hydroxide solution. After reacting for one hour, 50 cc. of N/2 hydrochloric acid solution are added and then titrated with N/2 sodium hydroxide, using phenolphthalein. The cc. of N/2 sodium hydroxide solution consumed multiplied by 6 yields the equivalent of aldehydes present and capable of oxidation to acids which are present in 300 cc. of the watery portion.

(3) Addition of the cc. of N/2 sodium hydroxide solution needed for the acid and aldehyde determination above furnishes a means of measuring and judging the total mixture of acids, and also substances capable of being oxidized to acids.

(4) The volume of the water-insoluble portion furnishes an index of the unconverted oil still present in the final reaction product.

(5) The saponification number of the water-insoluble portion is a measure of the degree of oxidation of this fraction.

The results obtained are tabulated below. (50 cc. of petroleum and 150 cc. of water are vaporized, with 150 cc. of water added during the reaction.)

TABLE I

Test No.	Temp. °C.	Air 1/Hr.	Time Min.	Watery Portion CC of: Acid	Aldehyde	Total Acids	Water-Insol. Portion CC	Sap'n No.	Remarks
1.	370	45	120	9.0	0.0	9.0	40.0	2.1	
2.	400	45	120	21.0	3.6	24.6	40.0	2.8	
3.	440	45	120	27.0	11.6	38.4	38.0	2.1	
4.	470	45	120	14.7	4.8	19.5	41.0	0.7	
5.	440	45	90	12.0	2.4	14.4	45.0	1.4	
6.	440	90	90	10.2	1.8	12.0	45.0	1.9	
7.	400	45	120	3.0	0.0	3.0	42.0	1.4	On Treated Kerosene
8.0	440	45	120	8.7	0.0	8.7	42.0	0.6	
9.0	470	45	120	3.0	0.0	3.0	41.0	0.3	

Tests 7-9 inclusive were carried out on an acid-treated stock.

The results of the tests are graphically shown in

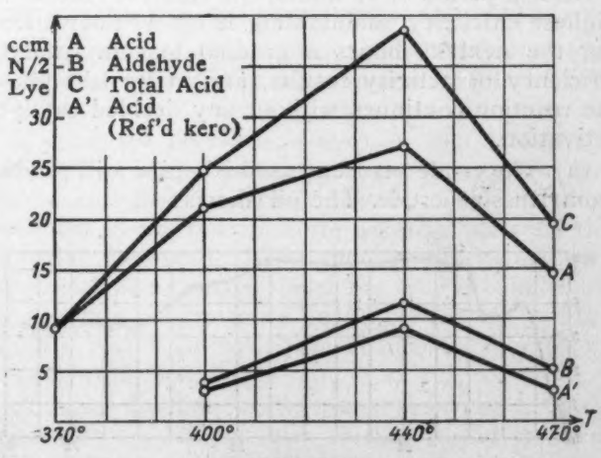


FIGURE 1

Figure 1, as acid, aldehyde and total acid formation. In Tests 1-4 inclusive a maximum is reached at 440° C. for acid-formation A, as well as for the aldehyde formation B. A maximum value (Nos. 7-9 inclusive) is also reached at 440° C. in the case of the acid-treated oil for acid-formation A′, although this value is considerably below that formed in the untreated stock. An untreated stock is therefore easier to oxidize than a refined product.[2]

Attention is also called to the fact that with the rise and fall of the acid and aldehyde formation there is also an attendant parallel rise and fall of the saponification number of the water-insoluble portion, which apparently reaches a maximum already at 400° C. (Table 1.)

Of peculiar interest is the entire absence of aldehydes in the case of the oxidized acid-treated stock. According to this, the aldehyde formation is related to the content of unsaturates, for instance, olefine hydrocarbons in the oil, which the acid treatment removes. The addition of a molecule of oxygen to the unsaturated residual valences of the olefines is probably a preliminary step to the formation of aldehydes; the olefines on further reaction, and with the rupture of the molecule at the double bond, give rise to aldehydes and finally acids.

On the basis of these preliminary results, large scale oxidations were next carried out under the conditions obtaining in Test 3.

In order to be able to test out larger quantities of oxidized material, greater quantities of kerosene were converted in a longer time period.

By such means 2075 cc. of kerosene and 6225 cc. of water were converted in 83 hours using 3735 liters of air. We obtained 1730 cc. of water-insoluble fractions, equivalent to a conversion percentage of 16.6 percent; 6170 cc. of water-soluble portion of a total acidity equal to 21 grams of acetic acid and 17 grams of solids were obtained containing 4.2 grams of actual acids. Basing the yields of water-soluble acids and the solid acids on the converted kerosene, one obtains 7.5 percent of converted material.

For large scale processing, the reaction tube was filled with fresh catalyst. The acids formed in the water layer during a definite time interval give a picture of the efficiency of the catalyst. The results are shown in Figure 2, where the titrations were conducted on 10 cc. of the water layer using N/10 caustic soda solution.

After a period of 15 hours, the catalyst reaches its highest efficiency, maintaining it for 30 hours. During the next 20 hours a gradual loss in catalytic efficiency or activity results, and in its last phase, the reaction continues without any decided catalytic activation.

(a) The crude kerosene used is a pipe still product from Russian crude. The oil shows:

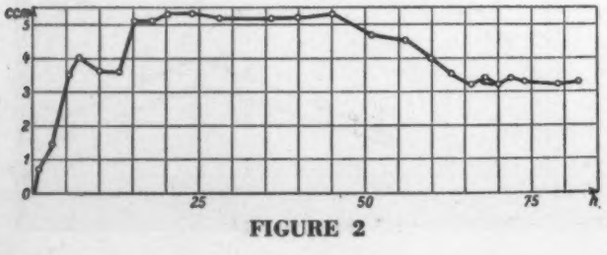


FIGURE 2

Flash—Over 30° C.
Pour—Minus 35° C.
Acid Value—0.026 percent, as SO_3.
Saponification Number—1.1.

The crude kerosene was neutralized with 5 percent of a 10 percent solution of lye, water washed, and dried over calcium chloride. This product showed:

Specific Gravity at 20° C. 0.8025
Refract. Index at 20° C. 1.4478

Engler Distillation Test:

I. B. P. ..... 178° C.
5 percent ..... 196° C.
10 ..... 203° C.
20 ..... 208° C.
30 ..... 214° C.
40 ..... 221° C.
50 ..... 227° C.
60 ..... 236° C.
70 ..... 244° C.
80 ..... 256° C.
90 ..... 268° C.
95 ..... 279° C.

Besides determining the above values of physical tests, a determination of the groups of hydrocarbons present should give a good idea of the chemical constitution of the kerosene used in this work.

The determinations of the unsaturated hydrocarbons was carried out by the rapid iodine value method of Margosches.[3]

The iodine value of 5.5 points to a very low percentage of unsaturates is present.

The estimation of the content of aromatic hydrocarbons was carried out according to the method of Hess[4], based on the principle that nitrating acid at low temperatures nitrates only the aromatics and these nitro-products are soluble in conc. sulphuric acid.

By this method the kerosene shows:

Aromatics ..... 12.2 percent

After the removal of the aromatics by nitration there remains a mixture of naphthene and paraffin hydrocarbons. The mixture shows:

Sp. Grav. 20° C. 0.7887
Refract. Index 1.4370
The ultimate analysis of this cut is:
Carbon ..... 85.5 percent
Hydrogen ..... 14.5 percent

The presence of the paraffin hydrocarbons undecane to tetradecane[5] can be concluded from the distillation test. By calculation of the average chemical composition of a mixture of these hydrocarbons and from the elementary chemical composition, the ratio of paraffins to naphthenes is found to be 20 to 80. Therefore, the proximate chemical composition of the kerosene is:

Aromatics ..... 12 percent
Paraffins ..... 18 percent
Naphthenes ..... 70 percent

(b) The water-insoluble Material.—The analysis of this cut follows the same lines as shown above; in the original product one finds:

Acid No. ..... 0.023% SO_3
Saponification No. 5.5
After neutralization and drying it shows:
Sp. Grav. 20° C. 0.8027
Refract. Index 20° C. 1.4470

Engler Distillation Test:

I. B. P.	189° C.
5 percent	203° C.
10	205° C.
20	209° C.
30	213° C.
40	220° C.
50	227° C.
60	234° C.
70	244° C.
80	254° C.
90	269° C.
95	279° C.

Other tests show:

Iodine Value (Margosches)	6.0
Aromatics (Hess)	12.0 percent

The mixture of naphthenes and paraffins after removal of aromatics shows:

Sp. Grav. 20° C.	0.7901
Refract. Index	1.4375

The ultimate analysis is:

Carbon	85.2 percent
Hydrogen	15.2 percent

From this one can calculate the relationship of paraffins to naphthenes as 40 to 60.

The proximate analysis of the water-insoluble portion is:

Aromatics	12 percent
Paraffins	35 percent
Naphthenes	53 percent

By the comparison of the above data, it appears that the Engler Distillation tests indicate that the low boiling point portions are oxidized first. The increase in the saponification number indicates that the formation of the higher fatty-acids also takes place, but to a lesser extent. It is of interest to note the shift in the paraffin-naphthene relationship, where the paraffins become relatively greater in the water-insoluble portion at the expense of the naphthenes. It would appear that the naphthenes are more easily oxidized than the paraffins. The greater specific gravity of the water-insoluble portion as compared to the gravity of the oil after aromatics removal can be explained as due to the removal of the lower B. P. portions by the oxidation.

(c.) The solid acidic substances separate out as a voluminous precipitate at the interface of both of the liquid reaction products. Filtration separates the precipitate. The greater amount of moisture can be removed by pressing it after which it is dried for four hours in a vacuum desicator; this results in a dry crumbly mass of dark color. It shows increasing solubility in the following solvents in the order named:

Ether,
86 Be'. naphtha,
Benzol,
Alcohol.

In all cases there remained, however, a brown powdery material which remained insoluble. By extraction with a 1:1 mixture of alcohol and benzol, the percent of insolubles was found to be 8.7 percent.

The solids separated above show:

Ash 2.9 percent
Spec. Gravity 20° C.—0.8905
Saponification No. 45.8
Acid No. 39.9
Iodine Value (Margosches) 3.7

The material does no show a definite melting point. White fumes appear when it is gently heated in a test-tube, the vapors condensing on the cool walls of the tube. The ash is red and partly soluble in hydrochloric acid. Iron was found in solution and silicic acid in the residue: the first derived from the reaction tube, and the second from the pumice carrier containing the catalyst.

From this data the composition of the solids obtained show them to be a mixture of 25 percent fatty-acids, and 66 percent unoxidized kerosene hydrocarbons, the remainder of 9 percent being impurities.

The percentage of solid acids[6] was calculated from the assumed molecular weight of 185, and by calculation from the saponification number, was found to be 25 percent. The acid number being practically as high as the saponification number indicates that the acids are free fatty-acids; the low iodine value indicates they are mostly solids.

(d.) The watery-portion.—This is a light brown liquid of very pungent odor. It is strongly acid, 50 cc. consuming 4.1 cc. of N/2 caustic soda solution (phenolphthalein.) For further analysis, a separation into single groups was next undertaken: 1000 cc. of the liquid were neutralized with 35 cc. of a 10 percent sol. of potassium hydroxide, using litmus indicator. A red brown precipitate resulted. This was filtered off, and identified as basic aluminum formate and acetate. Besides volatile aldehydes and ketones, calcium salts of the water-soluble acids that formed in the oxidation are found in the dark colored neutral filtrate. During distillation the volatile substances are now found in the clear pungent distillate No. 1 which amounted to 150 cc. Continuing the distillation, ordinary water distills off next, and the residue can be further concentrated to about 100 cc. By adding next, 35 cc, of dil. sulphuric acid (1:1) and again distilling, the bound fatty-acids are now set free and are collected in the receiver as distillate No. 2.

Due to the low concentration and the presence of many complex compounds, we were not able to obtain the individual groups in pure form for further identification or purification. One had to be content with proximate group analysis of the reaction product, and to be able to show the presence of individual components by the use of various and characteristic reactions or laboratory color tests. These reactions were tested out by the use also of pure chemicals as controls.

DISTILLATE NO. 1. ALDEHYDES AND KETONES

The various reactions characteristic of aldehydes and ketones were tested out, as enumerated in Table II, below.

To show the presence of ketones, distillate No. 1 was oxidized with alkaline hydrogen peroxide by the method of Blank and Finkenheimer. (1). the aldehydes are converted into the alkaline salts of the respective carboxylic acids, the ketones being inert and remain unconverted under the special conditions. The separation was carried out by distillation and the distillate was next tested according to the list of tests shown in Table III.

After the distillation and removal of the ketones, the distillation residue was further tested for propionic, butyric, valeric and capric acid according to

TABLE II
Special Aldehyde Reactions

AUTHOR	Reagent	Reaction	Characteristic For
Tollens (7)	Ammoniacal silver solution	Silver mirror	Aldehydes
Fehling (8)	Alkaline copper solution	Reduction	Aliph. aldehydes.
E. Fischer (9)	Phenylhydrazine chlorhydrate	Phenylhydrazone	Aldehydes
Caro (10)	Fuchsin-sulphurous acid	Red-violet coloration	Aldehydes, ketones, etc.
Bitto (11)	Alkaline sod. nitroprusside	Red coloration	Aliph. aldehydes and ketones with at least 2 C atoms
Barbet-Jandrier Istrati (12)	Phenol, conc. sulphuric acid	Red-brown ring	Aldehydes
Riegler (13)	Phenylhydrazine-NaOH	Rose red color	Aldehydes
Schryver (14)	Phenylhydrazine chlorhydrate; pot. ferricyanide, and hydrochloric acid	Fuchsi-red color	Aldehydes
Grosse-Bohle (15)	Fuchsin-sulphurous acid, hydrochloric acid	Violet color	Formaldehyde
Leys (16)	Mercuric oxide in solution in sod. sulphite and pot. carbonate	White precipitate	Acetaldehyde
Auld & Hantsch (17)	Mer. chlor and pot. hydroxid solution	White precipitate	Acetaldehyde
Melzer (18)	Sulphuric acid, phenol and alkalie	Negative	Absence of benzaldehyde
Schiff (19)	Anilineacetate	Negative	Absence of furfural
Rosenthaler (20)	Sod. nitrite, sulphanilic acid, and hydrochloric acid	Negative	Absence of alcohols

TABLE III

AUTHOR	Reagent	Reaction	Characteristic For
Tollens (21)	Ammonia silver solution	Negative	Absence of aldehydes
Caro (22)	Fuchsin-sulphurous acid	Red color	Ketones
E. Fischer (9)	Phenylhydrazine chlorhydrate	Formation of hydrazone	Ketones
Legal (22)	Sod. nitroprusside, alkalie, acetic acid	Claret red colors	Acetone
Reddelien (23)	Sulphuric acid; nitric acid	Negative	Absence of unsaturated ketones

TABLE IV

AUTHOR	Reagent	Reaction	Characteristic For
(25), (26)	Alcohol, sulph. acid	Odorous ester	Formic acid
Tollens (27)	Ammoniacal silver solution	Silver mirror	Formic acid
Weinland & Reihlen (28)	Iron chloride	Red coloration	Formic and acetic acids
(29)	Lead acetate	White precipitate	Formic acid
(30)	Dry distillation of the lime salts	Formaldehyde formation	Formic acid
(31)	Mercuric nitrate	White precipitate	Acetic acid
(32)	Sod. salt heated with arsenic trioxide	Cacodyl formation	Acetic acid
Agulhon (24)	Copper and iron salts with org. solvents	Negative	Absence of propionic, butyric, valeric, capric acids
(33)	Calcium chloride	Negative	Absence of oxalic acid
Deniges (34)	Hydrogen peroxide, iron chloride, acetic acid	Negative	Absence of benzoic acid

Agulhon,[24] but the presence of these was not proven. The aldehydes of these acids were also shown to be absent.

DISTILLATE NO. II. CARBOXYLIC ACIDS

When the alkali salts of the carboxylic acids are distilled with sulphuric acid, several milligrams of a white crystalline solid separated in the condenser and in the distillate recovered. This solid is insoluble in water, but soluble in alkali, and shows an indefinite melting point of 98-103° C. Further tests could not be made on this small amount of material. The reactions used to verify carboxylic acids are shown in Table IV., below.

The analysis of the watery portion indicates that aldehydes and ketones are formed besides the lower fatty-acids. In single fractions one could identify formic acid, and formaldehyde as well as acetic acid, acetaldehyde and acetone.

SUMMARY

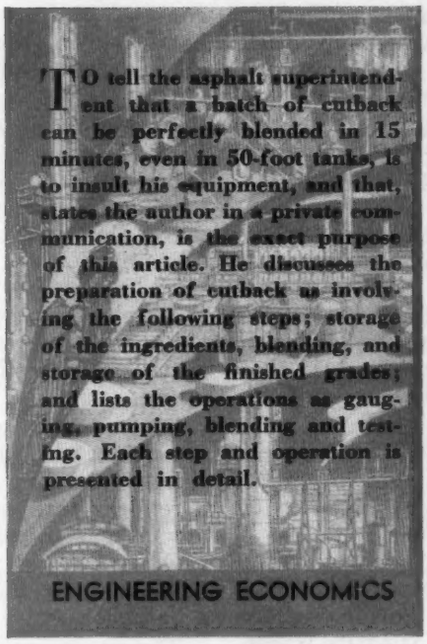
The optimum working conditions for the oxidation of a naphthene base Russian kerosene were determined when employing an apparatus described in Petroleum Zeit. Vol. XXX, No. 13, 6, (1934). It was required to obtain the best yields of water-soluble substances by catalytic vapor-phase air oxidation of this oil. Vanadic acid catalyst was employed (5 percent deposited on granular pumice stone.)

There were obtained:

(*Continued on page 190*)

Asphalt Cutting

GORDON MacLEAN
Turbo-Mixer Corporation



TO tell the asphalt superintendent that a batch of cutback can be perfectly blended in 15 minutes, even in 50-foot tanks, is to insult his equipment, and that, states the author in a private communication, is the exact purpose of this article. He discusses the preparation of cutback as involving the following steps; storage of the ingredients, blending, and storage of the finished grades; and lists the operations as gauging, pumping, blending and testing. Each step and operation is presented in detail.

TO TELL the asphalt superintendent that a batch of cutback can be perfectly blended in 15 minutes, even in 50-foot tanks, is to insult his equipment, and that is the exact purpose of this article.

Asphalt cutbacks have made a place for themselves which accounts for thousands of tons of petroleum products each year but, like all other asphalt operations, it has been one of the backyard operations of the refinery. Relegated to the far corner of the refinery, it had to be content with second-hand equipment of all sorts. The men in the asphalt division were the forgotten men.

New equipment came to the main gasoline production line, to be shortly replaced by still newer equipment. Lube production went through frequent upheavals, but the asphalt plant still pegged along with its discarded horizontal stills, battered tanks and used pipe.

This picture did not hold true in a few refineries, and there is no question but that many others now are paying more attention to this part of their business. It is certainly true, however, that the asphalt plant, dealing as it does with the dregs of the main processing line, has been treated like the proverbial step child.

Here and there, throughout the country, modernization has set in with results that have been surprising, 10-hour blending times reduced to 30 minutes or less, storage tanks which give uniform samples from any point, and continuous blending at the rate of 200 barrels an hour or more. These developments all spell progress, and for the refinery in the financial position to install them, mean that there is a recognition that asphalt products can be made to pay real profits.

The operations involved in making cutbacks are simple and, where the equipment is adequate, the control can be made equally simple. In general the cutting of asphalt involves only the storage of the separate ingredients, blending, either batch of continuous, and storage of the finished grades, and the operations may be listed as, gauging, pumping, blending and testing.

Simple operations, all, but there must be unvarying accuracy or the work must be done over, for specifications must be met to assure acceptance of the material. Inaccuracy or poor methods in any one of the four operations will cause endless trouble.

Cases have been known where it took four days to get the material blended and adjusted to the point where specifications could be met. Such trouble is a definite indication that the equipment is inadequate, resulting in a wholesale waste of time, and possibly the loss of a contract. By the time such a batch is finally acceptable, nerves are ragged and there is even a serious lack of faith in the final results.

With such a simple sequence of operations and with no unexplainable chemical reactions to complicate matters, the cutback plant should be the smoothest running part of the refinery, and it can easily be brought to that position. The simplicity of the operations should almost assure this, but there can be interfering factors at many points.

Gauging comes first for it is of even more importance in making cutbacks than it is in the gasoline department. It stands to reason that if the characteristics of the materials to be blended are accurately known, the results of the blending can be predicted, at least closely enough so that not more than one adjustment is needed.

Gauging involves two factors, volume and temperature, since the latter materially effects the former. The more accurately both are read, the better are the chances of getting the OK on the batch by the required shipping hour. Since the volume varies with temperature, correction tables should be avail-

able so that all figuring can be done at standard conditions, and the results should be checked by graphic charts so as to avoid decimal point trouble.

With gauging goes sampling, and here, again, is an operation that has been an integral part of refinery work since the beginning. In asphalt work, where viscosities are on a higher level, the problem of getting an absolutely representative sample to the laboratory is of unquestioned importance. If the final blend is going to be based on the laboratory results, it is undoubtedly wise to do the testing on a sample which accurately represents the entire batch to be used in making the cutback.

Right at this point, however, lies much of the explanation for "unexplainable" deviations from specifications after the initial blending. Conformity to specifications cannot be expected unless there was uniformity in the supply tanks during sampling. If the feed to the supply tank is of variable quality, the need for mixing in these storage tanks is obvious.

The point that is being emphasized here is that, with a previous knowledge of how the final physical constants of a cutback are effected by the proportions of asphalt and solvent, there is only one reason as to why the final specifications cannot be met with only one minor adjustment, and that reason is that gauging and sampling is done on tanks, the contents of which are not uniform.

Uniformity, in the case of large storage tanks, may be harder to attain than with small ones, but it is still necessary. Air blowing has been resorted to to accomplish this, but even in the refineries where it is being used every day, no one has a good word for it. It gives the appearance of marvelous action on the surface of the liquid, but on the bottom, where the agitation should be at its best, it is at its worst.

Where high-pressure air is used, blown through small holes, the original velocity is considerably higher and consequently more entrainment takes place at the bottom. Results show, however, that even this high-pressure air cannot give either the rapid blending time which is often desirable, or the assured uniformity which is always necessary.

It is obvious that the long blending time of air blowing is due to poor mixing action. Either the light constituents cannot be pulled down from the surface to blend with the bulk of the cutback, or some of the asphalt cannot be moved off the bottom and away from the corners. Uniform samples certainly cannot be drawn from a stratified tank, and without uniform samples the adjustments only postpone the shipment.

There are very much better methods of mixing available, methods which give much lower power costs as well as much faster blending. In analyzing the requirements for mechanical mixers for this operation, the problem of getting asphalt off the bottom of the tank, and getting the solvent down from the top, must get first attention. If the asphalt can be prevented from ever getting to the bottom, as a layer, and the solvent from getting to the top, the worst of the problem is solved immediately.

If, in addition, the bottom of the tank is swept by a rapidly moving horizontal stream, and a vertical mixing effect is assured, strong enough to depress the solvent and raise the asphalt, uniform samples should be secured within a few minutes of the starting time.

The fastest and most thorough equipment for this purpose, from the data at hand, is a type equipped with three centrifugal turbine type impellers on the same vertical shaft. The horsepower is low, and taking a probable mixing time of fifteen minutes into consideration, the horse-power-hours for blending large batches reaches a ridiculously low figure.

For blending operations, it is necessary to move large volumes of liquid, and this type of equipment may actually pump, through the three impellers, as much as 90,000 gallons per minute. If all parts of the tank are reached by the streams from the impellers, a running time sufficient to circulate the entire tank contents from 4 to 6 times will assure a uniform batch with a good safety factor. Samples taken from all parts of a tank so agitated will be uniform.

If the solvent is run in first, and the asphalt is then pumped in over the lower impeller, no free asphalt ever gets to the bottom of the tank, and solvent loss is decreased because it is an integral part of the cutback by the time it has become hot. By using this method, a sample may be taken only a few minutes after the last of the asphalt is in, for the blending has been going on during the filling of the tank. With such a process the burden of getting shipments out is thrown on the pumps, since the cutting and testing time is reduced to such a low figure.

The application of mechanical blenders to batch operation has been described, but the idea of continuous blending in small equipment, has lured many a refinery into trying it out. There are certain disadvantages to offset the obvious advantages of continuous cutting, and these are serious enough to have kept the bulk of production in the country on a batch basis.

The most serious of these disadvantages is that there is no positive assurance that the present flow is exactly like the sample taken 15 minutes before. No refinery would dare risk pumping the cutback from a continuous blender directly into tank cars. It would have to be pumped to storage and go through a final adjustment there, provided there had been any deviation in proportioning at any time during the run. In starting up such a system, there is almost sure to be some material which is not correct and this must be adjusted before it can be used, so the storage tanks must be equipped to agitate the material. Therefore it would probably have been simpler and quicker to have pumped to a batch blender in the first place. In a batch system, a slight variation in asphalt or solvent properties can be compensated for in the adjustment, but a continuous blender must be supplied with material of unvarying quality in order to operate at all.

Pump recirculation has been used for cutting asphalts, just as it is used for blending gasolines, but the time required for blending is just about proportional to the gallons recirculated, taking from 10 to 40 times as long to give uniformity as the high capacity inside impellers do. The pump horsepower is considerably higher, and the comparison of the required horsepower hours of the pump and inside impellers shows up in favor of the latter.

With the assurance that cutbacks can be turned out rapidly in any quantity, the necessity for large storage of finished goods is removed, which effects the amount of capital tied up and gives a degree of flexibility to the plant which is very desirable.

Revamped Treating System *Lowers Cost*

SHORTLY after beginning the manufacture of cracked gasoline at its plant at Allen, Oklahoma, Sunray Oil Company had some difficulty in reducing the cost of treating to a sweet and non-corrosive product. No difficulty was encountered in obtaining a sweet product, but the cost of chemicals used in the plant was too high. The unit in which the cracked gasoline was treated was built several years ago to treat straight-run gasoline from the atmospheric distillation unit.

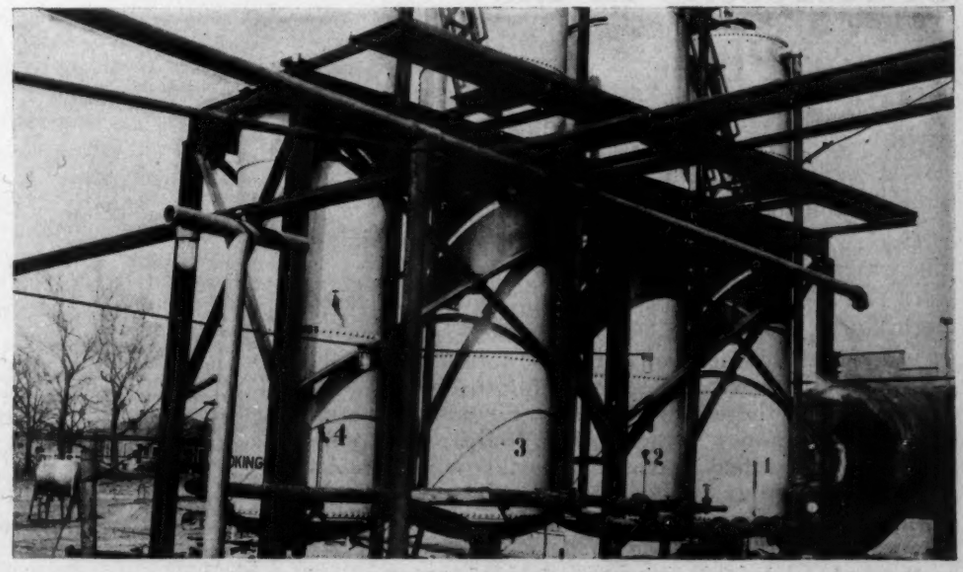
The treating plant consists of four columns set close to each other, supported by a framework of steel, and operated in a vertical position. When the plant was first constructed, the columns were not equipped with outside contacting nozzles, but depended upon the contact between the gasoline and the chemicals to occur within the column, accelerated by the use of perforated spiders through which the doctor solution was sprayed into the gasoline as it passed through the column.

As time passed, the drop in pressure between the chemical pump and the outlet of the chemicals through the perforated spiders continued to mount until it was with difficulty that an ordinary day's production could be processed. Upon opening the column to ascertain the difficulty, it was discovered that the spiders had become clogged, due to corrosion, and a deposition of scale and treating compound upon the pipes and within the orifices through which doctor solution passed. While the column was open, it was decided that much better contacting could be obtained if the spiders were removed, and contacting obtained through outside apparatus.

The plant was revamped, removing all spiders, and the first column placed in service for pre-contacting, or washing with a caustic solution to remove hydrogen sulfide. The second column was prepared as a receiving tank to contain the gasoline and doctor solution after contacting, while the third was to be used as a settling tank to remove doctor which might be mechanically carried over with the stream of gasoline. The third column is used for water washing so that no caustic will pass from the treating plant to storage and the inhibitor vessels.

Four-inch piping is used for transmission of gasoline into and around the four vertical vessels, with a connection made at the first tank for the introduction of caustic which is recirculated in the primary steps in treating the cracked distillate. Only a small amount of hydrogen sulfide is present in the gasoline as it comes from the receiving drums at the cracking unit, and consequently the caustic does not deteriorate rapidly. A similar connection in the four-inch header downstream from where the overhead line is attached removes the gasoline from the caustic pre-contacting tank, which is used to introduce the doctor solution in the stream of gasoline passing to the second vessel in the series.

Immediately following this connection, downstream in the four-inch header, three alloy cast iron contacting nozzles were placed in series to obtain adequate contact between the doctor solution and the pre-washed cracked gasoline. These contacting nozzles were separated from each other by inserting pipe spools, with companion flanges, about two feet in length. Shortly after the revamped treating unit was placed in operation, it was decided that perhaps more efficient contacting could be obtained by using four nozzles instead of three as originally designed. The middle contacting nozzle was then removed with its spools on each end and a double "Y" was made. The foot of the first "Y" was flanged to the first contacting nozzle, and the foot of the second was flanged to the third nozzle, which placed the



Sunray Oil Company, Allen, Oklahoma, treating system. Column 1 is pre-caustic wash, 2 is doctor contact and settler, 3 is settling column and 4 is the water wash column.

arms of both "Y's" meeting at a point where the second nozzle was originally placed. By inserting a contacting nozzle between each pair of opposing arms of the two "Y's," the unit was in condition for operation with the nozzles in series, yet with the central pair in parallel. The pressure drop through the nozzles was immediately lowered to a point considered normal for such installations, and treating can be carried on as rapidly as desired.

Settling out of the doctor solution passed with the gasoline to the column is obtained by gravity, as the entrance to the vessel is only a short distance above the lower head of the tank. Further separation is effected in the same manner by passing the gasoline through tank No. 3, which catches only a slight amount of chemicals due to the rapidity of settling in the second column. The gasoline is then washed with water, contacted with the gasoline through a nozzle similar in design to those used in the header leading to the second tank. It then flows by process pressure through an overhead line from the water-wash settling tank to the vessels where it is treated with gum inhibitor.

Only recently has the method been changed in mixing and preparing the doctor solution. Formerly it was carried at about 16° Be. At present, the strength of the solution is carried at 30° Be., which is said to be responsible for the rapidity of settling in the receiving tanks. By increasing the gravity of the solution by using a larger amount of caustic in the original mixture, the cost of chemicals has been reduced from about 6 mills per barrel to less than 2 mills.

The method followed in reducing the solidified caustic to a liquid state is similar to that used generally by other refiners, and is accomplished by steaming the contents of the drums into an open vat before adding litharge. A sulfur pot was placed on the gasoline header at a point where it was received from the caustic pre-wash tank so a split stream of the gasoline would dissolve the sulfur. Flowers of sulfur is dumped occasionally into the vat containing the doctor solution circulated through the treating plant. Checking with tests made in the laboratory has shown that this method of adding sulfur secures satisfactory reaction, and does not require any more time upon the part of the operator than an occasional filling of the sulfur pot.

Doctor solution is prepared in three vats, steamed and aerated for regeneration, and as a working tank for the chemical pump suction. When tests show that the doctor should be regenerated, the chemicals are removed from the plant and suction taken from another vat. Steam is used to maintain the desired temperature for removal of the undesirable sulfur compounds while blowing. Blowing also is continued in the working tank while recirculating the chemicals through the treating plant, which is said to lengthen the time that a charge of materials can be used without the need of withdrawal and regeneration.

Since the plant was revamped and the present method of treating instituted, the cost has dropped materially. Instead of consuming approximately 30 drums of solidified caustic monthly, only 3 or 4 drums are used. When the solution becomes thick, syrupy and black, it is now considered far better for treating than a freshly-made batch.

Duplex steam pumps are used to circulate the treating solution through the contacting nozzles and it is drawn from the columns, or vertical tanks, from the conical base. Adequate sample cocks were installed in the header before contacting and settling, as well as downstream so finished samples could be obtained for testing.

Catalytic Air-Oxidation of Kerosene in the Vapor Phase

(*Continued from page 186*)

(1). A water-insoluble layer, (Upper layer: Mainly consisting of unconverted kerosene.)

(2.) A watery portion, (Lower layer, containing the water-soluble oxidation products.)

(3.) A heavy white precipitate at the interface of both layers (apparently solid fatty-acids.)

Individual hydrocarbons or narrow fractions could not be obtained in purified form due to the low concentrations of these individuals, and due to the many various substances present; nor could single substances be identified as pure chemical compounds. An approximate group analysis had to suffice, and single compounds proved to be present by their various characteristic reactions with special reagent.

By such methods it was shown that in this type of oxidation aldehydes and ketones as well as the lower molecular weight fatty-acids were formed in appreciable quantities. From certain individual cuts one could show the presence of formic acid and formaldehyde, as well as acetic acid, acetaldehyde and acetone.

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A Carbon Monoxide Analyzer

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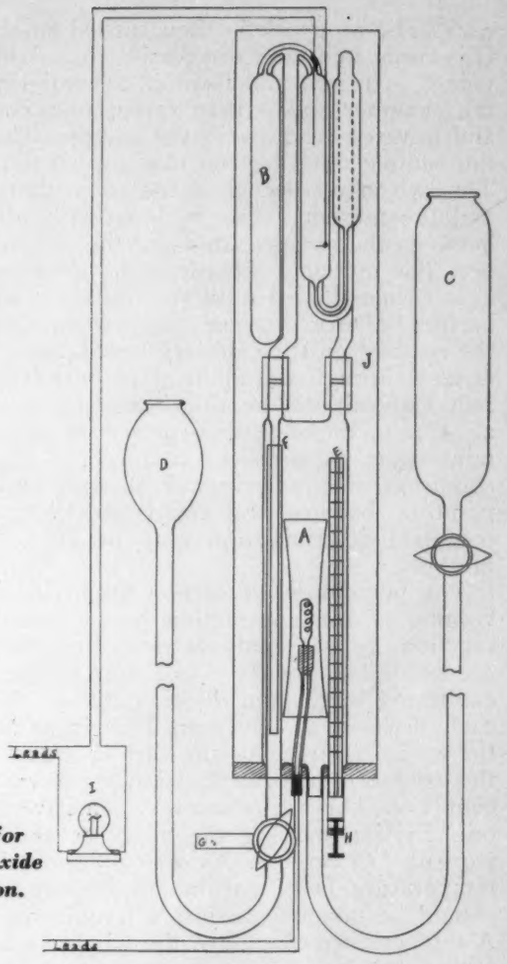
CARBON MONOXIDE determinations in air, flue gas or exhaust gas have hitherto required excessive time and care. A number of methods have been used, among which are: oxidation of CO to CO_2 and collecting in $BaOH_2$, conversion of H and CO to methane over a nickel catalyst, absorption of CO in blood to give characteristic spectrum, measurement of temperature rise when CO is oxidized, reduction of iodine pentoxide and measurement of released iodide, and by use of innumerable other oxidizing or reducing agents, absorbents, and catalysts.

By far the most popular of these methods is the iodine pentoxide method, in which the gas is dried, relieved of interfering hydrocarbons or organic matter, and is reduced by iodine pentoxide to release iodine. The iodine is absorbed and titrated. Although very accurate, this procedure requires much time in preparing the reagents, and in cleaning the system after runs. The iodine pentoxide requires four or five days for its preparation and purging before it is suitable for use in this apparatus. These preliminaries make a complicated and time-consuming process of this determination.

It was believed possible to construct an apparatus in which the carbon monoxide could be oxidized by combustion and the contraction recorded accurately enough to determine small amounts of the gas. This apparatus (after much designing and construction) was built.

As shown in the illustration, the combustion chamber, designated by A, contains the heating filament, around which is a circulating shield. When the filament is hot, the heated gas streams up through the shield, coming into contact with the hot wire in the restricted part. A constant and complete circulation is thus assured. Changes in volume are measured in the capillary E. This 1 mm. capillary is partially filled with mercury so that by adjusting the screw H to raise and lower the mercury column, a very sensitive volume adjustment may be made. The concentric tubes F are the means by which the combustion tube is connected with the compensator B. When the mercury reservoir D is lowered, the mercury level, clearing the bottom of the inner tube, permits connection between the chambers A and B. When raised again the mercury column cuts off connection between the compartments and yet permits some expansion, when the lower chamber is heated, without breaking the seal. By running mercury into the combustion chamber from reservoir C and then drawing it back into the reservoir, the gas sample is sucked in through G.

In order to measure the change in volume it is



Apparatus for carbon monoxide determination.

necessary to adjust the pressure in the combustion chamber to a zero point in connection with a reference pressure. The lower compartment is first balanced against the compensator and after combustion is again balanced. Thus, by maintaining a pressure equilibrium, the only variable factor is the volume. The compensator and manometer were first made in one unit with its combustion compartment. The two chambers were separated by a glass wall through which tubes made connection with the manometer and compensator. Concentric tubes formed the manometer. Because of difficulties of glass blowing and strains in the construction, this type was abandoned and the one shown was used. This compensator is similar to that designed by Martin Shepherd. The compensator is connected to the manometer by means of a capillary. Through F it is connected to the combustion chamber, which is permanently open to the other arm of the manometer. The contact points are of platinum. By using a 1.25 volt flashlight bulb, I, and a single flashlight cell, a good balance point indicator was obtained which did not foul the mercury by arcing.

The apparatus is surrounded by a water jacket. Temperature control is of greatest importance as even a very small temperature difference between the compensator and combustion chamber will cause volume errors. The apparatus might be immersed in a constant temperature bath, although this would detract from its portability. A jacket is therefore used with a stirring arrangement to maintain a conshtant current over all the parts.

To operate the analyzer the reservoir D is lowered until the mercury level is below the two-way stop-

cock. The stopcock is then turned so that the inlet G is connected with the combustion chamber. Reservoir C is raised and lowered several times to flush the chamber and is then raised to a constant mark and lowered to draw in the sample. The volume of the sample must be constant for all determinations. The two-way stopcock is turned so that G is cut off and the mercury rises in F to shut off connection between the compensator and the combustion chamber. The mercury column in the graduated capillary E is then adjusted until the indicator lamp shows a perfect balance between the two compartments, and the reading on the capillary noted. Sufficient current is sent through the filament to heat it to a medium red. Two minutes heating is usually enough to burn all carbon monoxide. A period of about 20 to 25 minutes is required for cooling the apparatus to a condition of equilibrium. E is then adjusted to the point of balance and the contraction recorded. A complete determination may be carried out in 30 minutes.

The percentage of carbon monoxide is twice the volume of the contraction in accordance with the reaction. In the analyzer described, 80 cc. samples are used. The capillary is 1 mm in diameter and is calibrated in 2.5 mm divisions. Thus the volume of each division is 1.96 mm. This is .0024 percent of the 80 cc. sample. As the carbon monoxide is twice the contraction, each division represents .0048 percent CO. The adjustment is sensitive to less than one division making the analyzer accurate to .005 percent CO or less. As was mentioned before, the temperature is of paramount importance and care should be taken to maintain it uniform throughout. A temperature change of the whole apparatus is immaterial, it being necessary only that all parts be at the same temperature when the balance point is determined.

In Figure 1 a cooling curve after combustion of an air sample is illustrated. The straight horizontal line indicates the point of balance before heating. The curve shows the lessening volume after the combustion as the chamber cools to a constant temperature. The curve should overlap the straight line at the point of equilibrium. The fact that it does not indicates either that combustibles were present in the air sample or, more likely, that more sensitive adjustments could not be made. As the difference is only .5 of a division we may take this as the representative accuracy.

TABLE 1
Analysis of CO in air.

Percent CO	Trial	Contraction	Calc. Percent CO
0.100	1	21	0.101
	2	20	0.096
0.050	1	11	0.053
	2	11	0.053
0.040	1	8.0	0.038
	2	8.5	0.041
0.030	1	6.5	0.031
	2	7.0	0.036
0.020	1	5.0	0.024
	2	4.0	0.019
0.010	1	3.0	0.014
	2	2.5	0.012

Figure 2 illustrates the cooling curve after combustion of a 0.1 percent CO in air sample. The difference or contraction as shown between the two lines is 20 divisions. This indicates the presence of .096 percent carbon monoxide.

Table 1 shows the results of analysis of samples or carbon monoxide in air. A standard sample of .1 percent CO in air was obtained and dilutions made down to .01 percent. The average deviation from the true values was .003 percent CO. This is about the optimum accuracy.

The applicability of this analyzer to air-carbon monoxide mixtures is evident. It is not necessary to dry the sample as a small amount of water should be kept in both compartments to neutralize errors due to vapor pressure.

In applying this method to carbon monoxide analysis in flue gas it may be seen that the results will depend upon the treatment of the gas before combustion. It has been shown that a liquid air trap followed by a chronic acid absorber in boiling water has been efficient in removing harmful constituents from gases in using the iodine pentoxide method. This treatment should also purify a flue gas sample for analysis in this apparatus.

Sufficient work was done with the analyzer on flue gases to show encouraging results. No experimenting on exhaust gases has been done but it is evident that the apparatus should be applicable to such analysis, with the proper precautions as to impurities.

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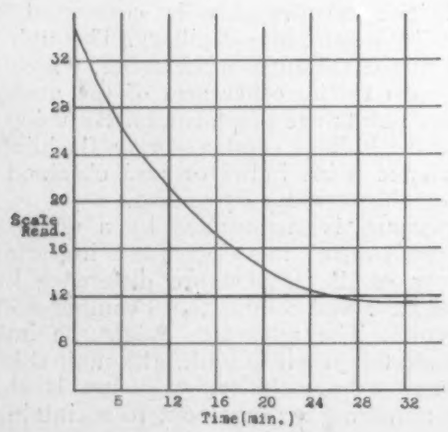


FIGURE 1
Combustion of air sample.

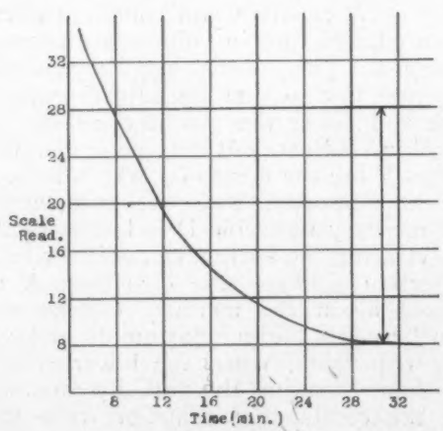


FIGURE 2
Combustion of .1 percent CO in air.

Drum dedenter, right foreground; paint stripper on the left, at The Texas Company's case and package plant, Los Angeles.

Straight Line Flow in Drum-Cleaning Plant

J. C. ALBRIGHT

WITH a drum-cleaning plant having a capacity of around 500 units each working shift, The Texas Company (California) reconditions, inspects and repaints every steel drum which is refilled with products. This reconditioning and repainting plant is located in the company's case and package plant at Los Angeles Harbor, where cargoes by water, car lots by rail and shipments by truck and trailer are handled. Drums are shipped and received by all these shipping agencies coming in as empties and reshipped filled with various products.

As the empty steel drums are received, they are stacked in the open yard near the drum cleaning building. An inspection is made to determine the physical condition of the packages, and if they are so dented and otherwise mis-shapen that they are unfit for refilling, they are discarded. Drums, however, that show dents and bruises which can be removed are passed immediately to the reconditioning equipment where they are reshaped to their original symmetrical appearance.

The first operation in reshaping and otherwise reconditioning the drums is through the dedenter machine. This apparatus is simple in arrangement and operation, being made in the form of a press just large enough for a standard oil barrel to fit nicely. The press is constructed of steel, having a base fitted with a raised section which fits snugly within the chime and upon which the closed head is to rest. The side members, the top and bottom sections are made of wide channel steel, all parts of which are securely attached to each other by welding. The upper section crosses from one riser to the other and contains a handwheel-operated threaded shaft to the lower end of which is fitted a head, and this, like the base, fits snugly within the chime of the upper end, but contains slots cut at the proper points to allow the open bungs to extend through.

A threaded connection which fits the bung of the drum is then attached and the drum filled with water through a hose leading from a pressure connection. When it is completely filled, pressure is placed inside

the drum until all minor dents and distortions are removed. An operator stands beside the press while pressure is being exerted within the drum, having a rubber mallet in his hand, which he strikes against the side of the drum near those dents which do not disappear with normal pressure. By judiciously using this hammer, all dents are made to disappear, and the drum resumes its original shape. The drum is then emptied of the water by siphoning it from the interior to a convenient disposal drain located beside the dedenter. A constant predetermined pressure is placed inside the drum while removing the dents, and because of this feature, no pressure which may rupture the drum is applied.

The next operation is the exterior cleaning unit which is located only a short distance from the dedenter. A raised track runs directly to the cleaning unit and is constructed of two pieces of wide angle iron, spaced at the proper distance so the rolling ridges on the side of the drum will come inside the angles of the track. This track slopes gently downward towards the exterior cleaning unit so the drum will roll by gravity to the entrance of the chemical vat. The dedenting unit operator removes the drum from his machine and places it upon the track, and upon a return trip places another on the unit for reconditioning.

The exterior cleaning unit is partly below the floor surface. The base of this unit consists of a steel vat which contains chemicals used to remove paint. Brushes are incorporated in this machine which come in contact with both heads and the outer surface of the drums, scouring the loose paint away so that paint still adhering may be more rapidly attacked by the chemicals in which the drum rolls while it is being cleaned.

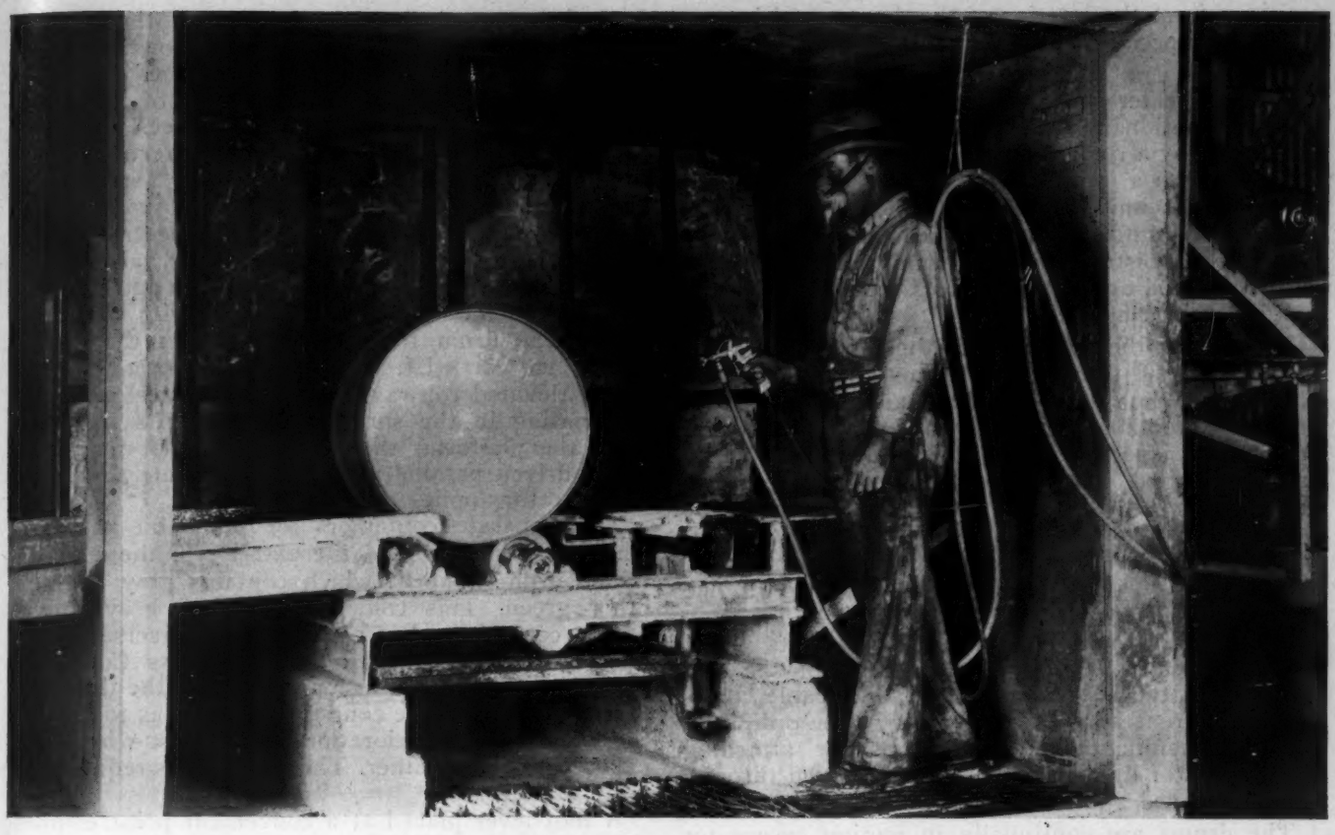
Clean chemical solution for exterior cleaning is placed in the vat before the start of day's operation, and solution is kept at a high temperature by means of a steam coil.

The cleaning unit is motor-driven, with the power unit placed upon a steel structure beside the vat, and is connected to the drum-rotating device by a series of chains and sprockets. The controls are on the side opposite to the motor, and are so constructed that the operator may allow a fresh drum to roll into the cleaner or remove the one just finished without touching any part of the machine except the levers and foot controls. An ingenious stop was built into the cleaning unit to prevent a drum from rolling into the vat except when the operator desires. Two flat sections of steel were attached to the rim of the chemical vat with their ends sharply bent to hold the drum on the runway at the edge of the vat, and are operated by a foot pedal which is linked mechanically so the ends may be opened at the desired time to permit the drum to move from the extreme end of the runway and drop upon the cleaning rolls.

Because of the fumes generated when the chemicals attack the old paint on the exterior of the drums, provisions were made to remove them to the atmosphere above the building through a stack. The stack rests upon an elongated pyramid cover, made of sheet metal supported from the steel roof joists of the building by steel cables. All seams in both the cover and the stack are made by crimping the sheet metal and soldering the joints.

No fixed time is allocated for each drum while in the cleaning vat, because the paint may be removed from one drum in much less time than that required for the next one. The operator of this unit watches

Inside drum washing operation: Operator at right is removing plugs and operator at left is siphoning excess.



Drum painting booth.

the progress of cleaning, and uses his own judgment as to the time necessary to completely remove the paint. As soon as the surface is clean enough to permit the drum to be moved to the next operation, the operator steps on a foot control which lifts the drum from the vat, and at the same time moves a lever which spreads the end cleaning brushes away from the drum. It falls through a swinging door on the side of the cleaning unit and comes to rest for a short time on the track, where the chemicals adhering to the surface are permitted to drain to the sump below.

The drum is rolled on an inclined angle iron track to the next operation. Drums at this point are given a hot-water rinse to remove the liquid chemical and to wash off any soft paint that may be clinging to the drums. An employe removes both plugs which were screwed tightly in place before the drum was given its bath in the chemicals, and moves the line of drums against the interior cleaning apparatus. This unit accommodates six drums at a time, and consists of two pipe lines laid horizontally upon grating places above a drainage sump in the floor of the building. These lines were fitted with nozzles located at the proper distance in order that the drum can be inverted above the lines with a nozzle in each opening in its head. Nozzles rotate so that chemical fluid is sprayed to all parts of the drum. As the line of drums stand upon the cleaning nozzles, an operator operates a quick open-and-shut gate in the chemical supply line which flushes the interior of six drums in one operation. Drums are first flushed with hot water to remove all free oil and debris; second, they are thoroughly washed with chemical solution; third, they are rinsed with hot water.

Chemical solution for interior cleaning is circulated from a storage tank to jets for spraying the inside of drums. Piping which carries the chemicals is laid upon the joists of the building, as are the steam lines which are used in maintaining the most efficient temperature in the chemical solution storage tank. Pumps are used to circulate the chemicals which takes a suction at a sump—which acts also as a settling basin—and delivers solution to the storage tank; another pump takes suction on the storage tank and delivers the solution to inside washing jets.

Each drum compartment is divided from the other by pipe rails and each compartment is fitted with a drum slide which permits the operator to change ends with the drum in a minimum amount of time. As they are removed from the interior cleaning rack, the end with the openings are uppermost so that the workman may siphon all excess fluid from the interior. The siphon was made by using small diameter piping arranged so a stream of water will eject the small amount of water left in the drum when it is removed from the cleaning nozzles. By filling the cleaning rack with six fresh drums after the previous number has been removed with the open ends uppermost, the operator has sufficient time to siphon the liquid from the inside of the cleaned drums while the next batch is being internally washed.

A double track with free-running rollers was laid in the line of drum travel so they can be passed through the drum cleaning shed with a minimum amount of labor. Besides these tracks the drying nozzles were placed, so that by removing the drums from the tracks and laying them down upon the floor, an opening in the head may be slipped over a drying nozzle. Heated air is used for removing the

moisture from the interior of the drums, and is prepared in a special furnace built into the drum cleaning shed.

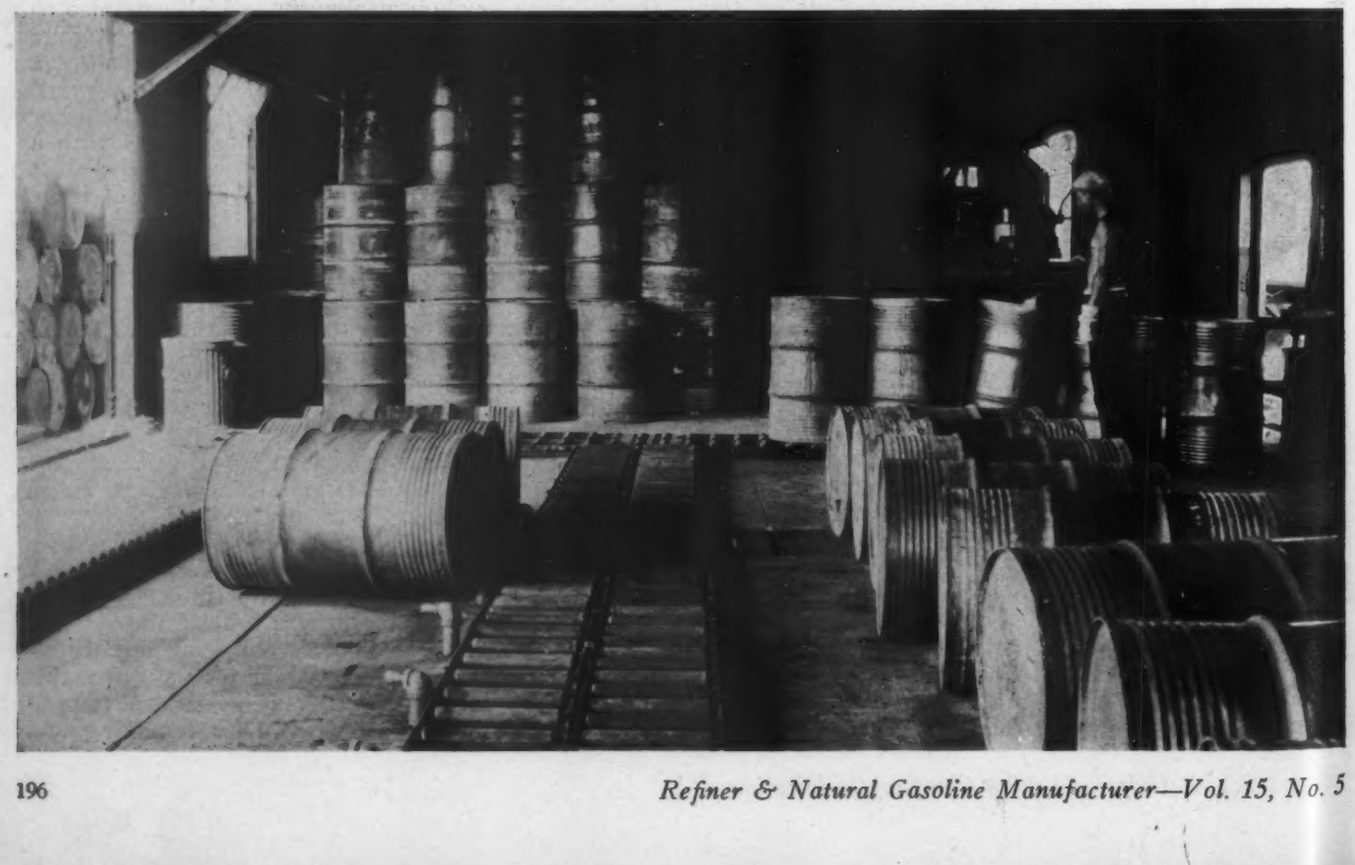
The air heater is similar to a rotary kiln, except that it is stationary with the air being forced into it at one end by a rotary blower and removed from the other through a pipe line which leads to the drying nozzles. This unit is gas-fired and the temperature of the air passing through the heater is sufficient to remove the moisture from the drums without becoming overheated and causing excessive fuel consumption in the heating element.

As soon as the moisture has been removed from the interior of the line of drums they are again placed upon the free-rolling track and passed farther down the building. At the end of this rollway the drums are inspected while in line of movement by an operator who inserts a rod terminating in an electric light by which he is enabled to determine whether or not the interior of the drum is in condition to be refilled. The inspection light consists of a hollow rod with a lamp socket attached at one end, insulated to prevent short circuits. The bulb used is one of the small sized lighting units which can be inserted readily in the larger of the two openings in the head of the drum. The cable carrying the current is of the heavy-duty type ordinarily used in manufacturing institutions for attaching hand lamps or light motors, and is threaded through the hollow rod and connected to the socket.

The drums are continually in motion except for the short time required for interior cleaning and drying, and after inspection, they are either stacked against the wall of the building, or placed on the rollway leading to the painting booth. This final inspection before painting is for both the interior condition, such as adhering scale and moisture not dried thoroughly, and for physical condition. If a drum has passed through the line of the various cleaning and drying operations and shows mechanical defects which might permit leakage, the drum is returned to the stack on the outside of the building and is discarded from further service.

The booth used for repainting the reconditioned drums is in one corner of the building near a door leading to the drum storage room. It is made of sheet metal with seams carefully made to prevent the sprayed paint from falling on other parts of the building. The front of the booth is open and has two windows on the opposite side in line with the drum travel. The drum to be painted enters the booth, after being carried from the final inspector's station on an elevated rollway, to drop on a steel platform terminating in the spraying position. As it comes from the platform, the drum is placed upon two motor-driven parallel rollers which rotate slowly as the operator applies the coat of enamel.

Two separate containers for the paint are used when applying paint to the exterior of the reconditioned drums, one of which contains gray and the other green. This color combination, gray drums with a central band of green, is the universal designation of drums shipped by The Texas Company. The application of paint, or enamel, to the drums is to make an attractive container, as well as to protect the drum from deterioration due to rust when it is exposed to the weather. The operator is required to wear a respirator.

A foot lever placed at a convenient point enables the paint operator to elevate the finished drum from the rolls to another inclined rollway which leads through the second small window and to the drying room where the paint is cured. Each drum as it is finished continues in the straight line flow through the heated room where the paint is dried and then enters the storage space to await being filled and stenciled.

Drum drying and inspection. Drying jets are shown in the foreground.